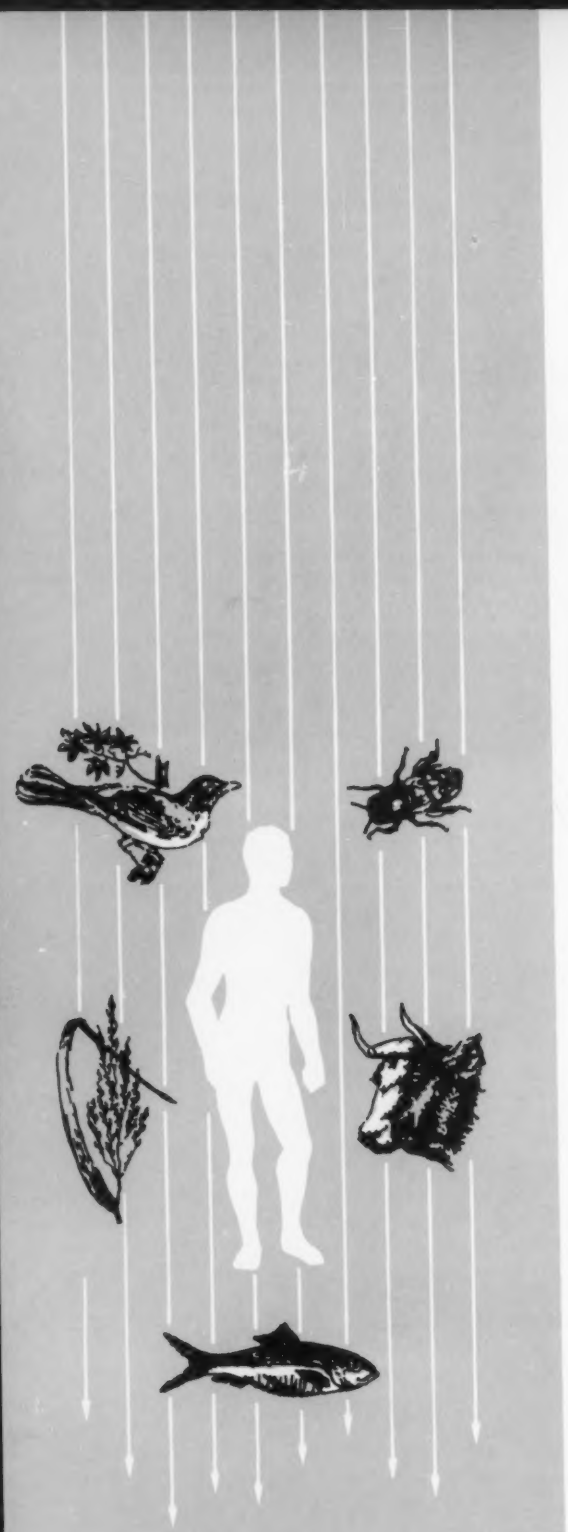

PESTICIDES MONITORING JOURNAL

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PESTICIDES IN PEOPLE

Effects of Reducing DDT Usage on Total DDT Storage in Humans

F. W. Kutz,[1] A. R. Yobs,[2] S.C. Strassman,[1]
and J.F. Viar, Jr.[3]

ABSTRACT

Agricultural uses of the insecticide DDT were cancelled by the U.S. Environmental Protection Agency December 31, 1972. However, the domestic use of DDT had begun to decline before this action. Beginning July 1969, residues of DDT and its metabolites were measured in human adipose tissue collected through an annual national survey. Levels of total DDT equivalent residues in human adipose have decreased slightly, but the frequencies of finding DDT or its metabolites have remained high. The most marked decline in residue concentration has been found in the youngest age group (0–14 years). Approximately 80 percent of the total DDT equivalent found in this survey was DDE. These data show that the reduction of the agricultural uses of DDT has decreased human exposure to and storage of this chemical.

Introduction

Agricultural uses of the insecticide DDT were cancelled by the U.S. Environmental Protection Agency (EPA) December 31, 1972. This cancellation order culminated approximately three years of intensive administrative inquiries into all uses of DDT. Most evidence indicated that continued general use of DDT represented an unacceptable risk to humans and their environment. The order did not, however, prohibit the continued use of DDT in controlled situations such as public health programs, quarantine programs, and other official uses in isolated instances (37 FR 13369).

Even prior to this action, DDT use in the United States had declined greatly (Figure 1). Peaking at approximately 80 million pounds in 1959, DDT use decreased to less than 12 million pounds by 1972. Since the 1972 ban, DDT has been used only in emergencies.

FIGURE 1. *Domestic use of DDT by year, 1950–72*
(D. L. Fowler, Control Stabilization and Conservation Service, U.S. Department of Agriculture, 1975: personal communication.)

Proof that DDT residues were present in human tissues collected over an extensive geographical area contributed to the cancellation decision. Considerable data on residue levels in humans in the United States were provided by the National Human Monitoring Program for Pesticides. This EPA program monitors residue storage, excretion, and metabolic effects in humans on a national scale in order to estimate the incidence and level of exposure to pesticides experienced by the general population and to identify changes and trends in this exposure.

This paper reports survey findings of total DDT equivalent during fiscal years 1970–74. The term, DDT equivalent, reflects the total burden of DDT and its analogs and compensates for differences in molecular weights of these compounds.

Methods and Materials

The Human Monitoring Survey during these five years collected samples of adipose tissue for chemical analysis. Tis-

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sues were obtained through cooperating medical pathologists at sites selected on the basis of an experimental design covering the conterminous 48 States.

SURVEY DESIGN

A proportionate, stratified-random sampling design was followed in selecting cities for sample collection. The population strata for the surveys conducted in fiscal years 1970–72 were the four census regions provided by the 1960 census; 39 collecting sites were used. The population strata for the surveys conducted in fiscal years 1973 and 1974 were the nine census divisions provided by the 1970 census; 75 collecting sites were used, an increase of 66 percent over the earlier survey. A fiscal year begins July 1 and ends June 30 of the year used for identification; i.e., FY 1970 extends from July 1, 1969, to June 30, 1970. The sampling units in each annual survey were cities with populations above 25,000 people. The number of sites needed in each area was based on the proportion of the national population in that area. Cities which served as collecting sites were selected at random.

The design provided samples which statistically represented the general population. For each collection site, an annual sample quota was established which reflected the demographic distribution in that census region or division. This quota in each group was proportioned according to age, sex, and race distribution of the population.

Samples were collected by cooperating pathologists and medical examiners from postmortem examinations and from specimens which had been removed during therapeutic and elective surgery. Thus tissues were received from patients with pathological conditions of varying severity and duration and from victims of sudden death from trauma or acute illness. Age, sex, race, and pathological diagnosis of the donor were recorded. Geographic residence was assumed to be the general location of the hospital. Since the objective of the program was to reflect pesticide levels in the general population, samples were not collected from patients with a suspected or established diagnosis of pesticide poisoning, from patients exhibiting cachexia, or from patients who had been institutionalized for extended periods. Further details of the program have been presented by Yobs (*6*) and Kutz (*1*).

CHEMICAL ANALYSIS

Samples were analyzed chemically by contract laboratories using methods specified by the program. Laboratories were required to maintain acceptable performance levels of an interlaboratory quality assurance program established and moderated by the EPA Pesticides and Toxic Substances Effects Laboratory, Research Triangle Park, N.C. The laboratory also provided technical assistance for the analytical portion of the program.

Samples were analyzed for selected chlorinated hydrocarbon insecticides using a modified Mills-Olney-Gaither procedure (*5*). A 5–g sample of human adipose tissue was dry macerated with sand and disodium sulfate and the lipid material was isolated by repetitive extractions with petroleum ether. Pesticide residues were extracted from the lipid material with acetonitrile and then partitioned back into petroleum ether by aqueous dilution of the acetonitrile extract. The petroleum ether extract was concentrated to 5 ml by Kuderna-Danish evaporation and transferred to a florisil column for successive elutions with 6 percent and 15 percent ethyl ether / petroleum ether. The eluates were concentrated in Kuderna-Danish evaporators and the final extracts were examined by electron-capture gas chromatography. Recovery studies indicated that all DDT isomers and analogs were recovered completely, above 80 percent. Thus the data were not corrected for recovery. Confirmatory techniques included thin-layer chromatography, Coulson detectors, microcoulometry and, in some cases, combined gas chromatography / mass spectrometry.

COMPUTATION AND DESCRIPTIVE STATISTICS

Residue values presented in this paper were calculated on a percent-lipid basis. Conversion was made for each specimen by dividing the whole tissue (wet-weight) value by the proportion of lipid extractable material. This method of reporting reduced the inherent variation in residue data which resulted from differences in the lipid content of individual adipose tissue specimens.

Data were characterized by several statistical parameters: sample size or total number of samples analyzed, frequency as percent positive, and geometric mean.

In calculating the descriptive statistics, only quantifiable amounts of pesticides were considered; trace amounts were converted to zero prior to data processing. Since these residue data were not normally distributed statistically, the geometric mean was considered a reliable measure of central tendency.

Results and Discussion

DDT is stored in greater amounts than are other organochlorine pesticides and is found in almost every adipose tissue sample analyzed (*1*).

DDT is either dechlorinated in the human body to TDE and then metobolized to the water soluble and excretable DDA, or it is excreted directly as DDT. DDE storage is not appreciably derived from ingestion of DDT, but rather by intake of DDE previously degraded in the environment from DDT. Since DDE is not effectively eliminated from the body, the result is a gradual increase in the body burden of this chemical. On the other hand, DDT is broken down and excreted more rapidly than DDE and thus is more responsive to changes in exposure level than are the other analogs (*3, 4*).

The annual geometric mean levels of the national survey and the data stratified by age group are presented in Table 1. Sample size and frequency of detecting total DDT equivalent residues are shown in Table 2. The data indicated a nationwide decrease in total DDT equivalent residue concentration. A more marked decrease in concentration was apparent in the youngest age group (0–14 years). Residues decrease noticeably in all age groups when data are stratified by age group. The frequency of detecting DDT and its transformation products in human adipose tissue has remained high, practically 100 percent in all groups and years.

Studies by Kutz et al. (2) showed that residues of DDT and its analogs are racially stratified. Samples from Negroes contained almost twice as much total DDT equivalent residues as did samples from Caucasians. In light of this fact, data in the present study were further stratified by race and age group (Table 3). The five-year trend toward decreasing levels of total DDT equivalent was still apparent for each racial group. Samples from Negroes collected in FY 1971 showed higher levels than did those in FY 1970, but the trend toward decrease compensated for this slight rise over the longer time period.

Conclusions

The concentration of residues of total DDT equivalent in human adipose tissue has decreased slightly; however, the frequency of finding DDT and its analogs has remained high. The slight decline in concentration may be due to reduced human exposure to DDT as a result of decreased use.

TABLE 1. *Total DDT equivalent residues in human adipose tissue from general population, United States*

Age, Yr.	FY 1970	FY 1971	FY 1972	FY 1973	FY 1974
	Residues, ppm Lipid Weight				
0–14	4.47	3.74	3.03	2.63	2.32
15–44	7.53	8.29	7.23	6.23	5.46
45 and above	8.88	8.64	7.96	7.13	6.97
National Summary	7.88	7.95	6.88	5.89	5.02

NOTE: Total DDT equivalent = (o,p'-DDT + p,p'-DDT) + 1.114 (o,p'-TDE + p,p'-TDE + p,p'-DDE + o,p'-DDE).

Residues expressed are geometric means.

TABLE 3. *Total DDT equivalent residues in human adipose tissue from general population by race, United States*

Age, Yr.	FY 1970	FY 1971	FY 1972	FY 1973	FY 1974
	Residues, ppm Lipid Weight				
	Caucasians				
0–14	4.16	3.32	2.79	2.59	2.15
15–44	6.89	6.56	6.01	5.71	4.91
45 and above	8.01	7.50	7.00	6.63	6.55
	Negroes				
0–14	5.54	7.30	4.68	3.16	4.02
15–44	10.88	13.92	11.32	9.97	9.18
45 and above	16.56	19.57	15.91	14.11	11.91

NOTE: See Table 1 for definition of total DDT equivalent.
Residues expressed are geometric means.

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TABLE 2. *Sample size and frequency of detecting total DDT equivalent residues in human adipose tissue, United States*

	FY 1970		FY 1971		FY 1972		FY 1973		FY 1974	
	Sample Size	Percent Positive	Sample Size	Percent Positive	Sample Size	Percent Positive	Sample Size	Percent Positive	Sample Size	Percent Positive
Age, Yr.[1]										
0–14	134	100	133	100	228	100	155	100	194	100
15–49	476	99.78	560	99.64	602	100	405	100	333	100
50 and above	802	100	921	99.78	1090	99.90	532	100	371	100
National Summary	1412	99.83	1616[2]	99.75	1920	99.95	1092	100	898	100

NOTE: See Table 1 for definition of total DDT equivalent.
[1] Age breakdown for FY 1973 and FY 1974 was: 0–14, 15–44, and 45 and above.
[2] Summary of sample size in FY 1973 and FY 1974 reflects addition of two individuals whose ages were not known.

RESIDUES IN FISH, WILDLIFE, AND ESTUARIES

Mirex Residues in Bobwhite Quail After Aerial Application of Bait for Fire Ant Control, South Carolina—1975–76 [1]

Ronald J. Kendall,[2] R. Noblet,[2] J. D. Hair,[2] and H. B. Jackson [3]

ABSTRACT

*Mirex, the organochlorine compound used for control of the imported fire ant (*Solenopsis invicta *Buren), was applied aerially under supervision of the South Carolina Plant Pest Regulatory Service in October 1975 to a game management area in Hampton County, S.C. Influenced by recent reports indicating that low levels of mirex were toxic to certain nontarget organisms in laboratory studies, authors initiated a program for monitoring mirex residues in bobwhite quail (*Colinus virginianus*). Pretreatment residues were recorded on a dry-weight basis in bobwhite quail breast and adipose tissue; conversion factors for determining wet-weight concentrations are approximately as follows: fat, 0.77; and breast, 0.29. Residues ranged from 0.000–0.178 ppm and 0.247–2.763 ppm, respectively. Mirex residues in quail adipose tissue showed up to five-fold increase within the first month after treatment and declined thereafter.*

A residue peak was noticed the spring following mirex treatment, corresponding with insect emergence. Mirex residues in quail collected in summer 1976 following a fall bait application showed slightly higher residue levels than had birds taken in summer 1975; however, little, if any, human food chain contamination would result in the consumption of birds with residue levels found in this study.

Introduction

Mirex, a polycyclic chlorocarbon, is the toxic ingredient in the bait used to control the red imported fire ant (*Solenopsis invicta* Buren) in the southeastern United States. The bait is generally applied aerially in a dosage prescribed by the U.S. Department of Agriculture (USDA) (*12*) to infested areas and becomes available to foraging fire ants. Nontarget species contact mirex by direct consumption of the bait, by scavenging on contaminated carcasses of poisoned ants, and/or by consuming organisms which have concentrated mirex from the environment (*13*).

Mirex has an affinity for lipids and, like certain chlorinated hydrocarbons, is stored and accumulated in the adipose portion of animal tissues (*10*). The persistent nature of mirex is indicated by Kaiser, who detected mirex in fish from Lake Ontario (*9*); however, the contamination source was probably a chemical manufacturer which produced mirex in that area. The movement and accumulation characteristics of mirex are responsible for the widespread presence of this chemical in animal life, especially in avian predators (*3*).

In monitoring mirex residues in nontarget organisms after treatment, Naqvi and de la Cruz (*13*) noted that concentrations of the pesticide in various trophic levels indicated biological magnification. Baetcke et al. (*1*) found significant levels of mirex in wildlife collections one year after treatment; bobwhite quail (*Colinus virginianus*) had up to 3.148 ppm mirex in adipose tissue.

The proposed mirex treatment area had a severe fire ant infestation while serving as game management land for bobwhite quail hunting. The authors wanted to measure residues in quail available for human consumption because the birds' food habits (*14*) allow frequent contact with mirex. Therefore, this study was initiated to monitor mirex residues in bobwhite quail before and after treatment.

Methods and Procedures

SAMPLE AREA AND APPLICATION OF MIREX

Mirex bait (1.7 g mirex/acre in 1.25 lb. corncob grit/soybean oil bait) was applied aerially to the James W.

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Webb Wildlife Center, Hampton County, S.C., under supervision of the South Carolina Plant Pest Regulatory Service (SCPPRS). This area is managed by the South Carolina Wildlife and Marine Resources Department (SCWMRD). A 1400-acre tract, excluding the ponds, of the 6000-acre quail plantation was treated. Various habitats were represented in the study area, including pine stands, hardwood forest, broom sedge fields, and farms. Application and collection dates are listed in Table 1.

SAMPLE COLLECTION

The SCWMRD permitted as many as eleven pretreatment quail and five posttreatment quail to be taken from the sprayed area each month. The bobwhites were shot, labeled, and transported to the laboratory on ice and frozen at 0°C until they were thawed and various tissues were removed.

EXTRACTION

Two tissues, fat (50–200 mg) and breast muscle (200–500 mg), were sampled from each bird, weighed, freeze-dried, weighed again, and ground with mortar and pestle using granular sodium sulfate as an abrasive. Mirex was extracted from breast muscles with hexane and from fat samples with petroleum ether. After grinding, the samples were collected in 125–ml flasks and fat samples were lightly boiled on a steam bath for 1 minute. All samples were then shaken on a wrist-action shaker for 1 hour.

CLEANUP

Extracts were cleaned by using standardized florisil column chromatography. Ten cm of 60/100–mesh PR grade florisil topped with 2 cm of anhydrous sodium sulfate was placed in a 1-cm-ID glass column fitted with a fritted glass disc. The florisil had been activated by oven heating at 150°C for 3 hours.

Columns were prewashed with 40 ml petroleum ether, and the washings were discarded. The extract was filtered through Whatman No. 40 paper to remove tissue debris, placed on the column, and allowed to percolate into the florisil; then the mirex was eluted with 40 ml of 6 percent ethyl ether in petroleum ether. The columns were stripped with 40 ml of a 15 percent ethyl ether in petroleum ether solution before introduction of the next sample. Three samples were cleaned on each column. The eluants were collected in 80-ml glass beakers, evaporated to dryness on a steam bath, and transferred into aluminum-lined cap sample vials with 5 ml of nanograde hexane. Stock solutions were adjusted from 0.5 to 5 ml depending on the concentration of mirex.

TABLE 1. *Dates of mirex application and bobwhite quail collection in South Carolina game area, 1975–76*

PRETREATMENT SAMPLES: AUGUST 22-SEPTEMBER 30, 1975	
BAIT APPLIED: OCTOBER 7, 1975	
14 day posttreatment sample:	October 21, 1975
28 day " "	November 4, 1975
42 day " "	November 18, 1975
74 day " "	December 20, 1975
100 day " "	January 17, 1976
145 day " "	March 1, 1976
235 day " "	May 30–June 6, 1976
280 day " "	July 15, 1976

QUANTIFICATION AND CONFIRMATION

Mirex residues were detected with a Micro-Tek 220 gas chromatograph using the folowing instrument parameters and operating conditions:

Detector: Ni-63 electron-capture
Columns: 6 ft by 1/4 in., U-shaped glass packed with mixture of 10 percent DC–200 on 80–100 mesh Gas-Chrom Q and 1.5 percent OV–17, 1.95 percent QF–1 on 80–100 mesh Gas-Chrom Q

Temperatures:	
injection port	220°C
column	210°C
detector	300°C

Carrier gas: nitrogen, flowing at 100 cc/min; purge flow, 10 cc/min

Five-ml aliquots were injected into the gas chromatograph using a Hamilton 10 μl syringe. The detection limit of mirex in tissue samples was set at 0.01 ppm. If mirex was detected in quantity <0.01 ppm it was recorded as a trace. Recovery tests were performed but values given here were not corrected for recovery. The recovery rate for mirex in tissue samples was approximately 70 percent. All concentrations of mirex were reported on a dry weight basis. Mirex was confirmed in several samples by mass spectroscopy.

Results and Discussion

Table 2 presents mirex residues in fat and breast tissue of 48 bobwhite quail according to sex, age, and time of collection.

Mirex residues were detected in pretreatment samples of bobwhite quail. Although the Webb Center had not been recently treated with mirex, the area bordering the plantation was treated by SCPPRS the spring before pretreatment collections of quail. Therefore, some mirex residues were anticipated.

Adult male bobwhites were selected for pretreatment collection because authors believed that they were more likely to contain high residues than females (Kendall et al., 1976: unpublished data). The pretreatment data ranged from 0.000–0.178 and 0.247–2.763 ppm mirex in breast muscle and adipose tissue, respectively. Baetcke et al. reported similar mirex residues in adipose tissue (0.000–3.148 ppm) of bobwhite quail collected 1 year after a mirex treatment (*1*).

Random posttreatment samples of young and adult bobwhite quail were collected (Table 2). The mean residue level of mirex in adipose tissue had up to a five-fold increase 2 weeks after treatment; however, this must be interpreted carefully, due to age variation in the birds. Juvenile quail apparently accumulated mirex quickly after bait application, probably because young quail consume a

TABLE 2. *Mirex residues in fat and breast samples from bobwhite quail collected on South Carolina game area, 1975–76*

Sample [1]	Sex	Age [2]	Residues, ppm dry weight		Residues, ppm dry weight $\bar{X}$	
			Breast	Fat	Breast	Fat
Pretreatment	M	Adult	0.178	2.763		
"	"	"	0.045	0.521		
"	"	"	0.034	0.255		
"	"	"	0.173	0.816		
"	"	"	0.046	0.247		
"	F	"	0.000	0.453		
"	M	"	0.025	0.544		
"	—	"	0.000	0.695		
"	M	Adult	0.081	0.886		
"	"	"	0.000	1.212		
"	"	"	0.000	2.733	0.053	1.011
Posttreatment 1	M	Subadult	—	0.211		
"	—	53	0.499	7.705		
"	F	Adult	0.370	5.602		
"	—	45	0.018	13.631		
"	F	95	0.179	1.501		
"	—	65	0.141	2.231	0.231	5.117
Posttreatment 2	M	69	0.239	1.479		
"	F	Adult	0.035	1.188	0.137	1.330
Posttreatment 3	"	"	0.039	2.735		
"	"	110	0.087	1.484		
"	M	110	0.057	1.839		
"	F	Subadult	0.089	2.878		
"	M	89	0.170	4.730	0.088	2.733
Posttreatment 4	"	Subadult	0.057	1.655		
"	"	"	0.026	1.516		
"	F	"	0.056	1.629		
"	"	"	0.093	6.627		
"	M	"	0.085	5.485	0.063	3.382
Posttreatment 5	"	" [3]	0.000	0.000 [1]		
"	F	"	0.050	2.902		
"	M	"	0.063	2.383		
"	F	Adult	0.287	2.309		
"	F	Adult	0.034	0.477	0.109	2.018
Posttreatment 6	"	Subadult	0.089	0.980		
"	M	"	0.363	11.819		
"	"	"	0.123	4.975		
"	"	"	0.355	8.097	0.233	6.468
Posttreatment 7	F	"	0.023	0.284		
"	M	"	0.156	0.867		
"	"	"	0.125	3.631		
"	"	Adult	0.053	0.502		
"	"	Subadult	0.037	1.566	0.079	1.370
Posttreatment 8	M	Subadult	0.015	0.400		
"	F	"	0.028	3.843		
"	M	Adult	0.024	1.447		
"	"	"	0.042	0.886		
"	"	"	0.117	10.012	0.045	3.318

[1] See Table 1 for schedule of mirex applications and quail collections.
[2] Aged by method of Rosene, 1969; literature reference *14*: Juveniles 1–150 days, Subadults > 150 days, Adults > 270 days.
[3] This sample was a banded-released pen reared bird which had been in the study area approximately 2 weeks before collection.

much greater proportion of animal matter than do adult birds (*6*, *14*).

Furthermore, Naqvi and de la Cruz (*13*) reported mirex residues up to 0.7 ppm in grasshoppers and crickets collected from areas receiving a mirex bait application. Examination of crop contents of Posttreatment 1 birds revealed grasshoppers to be a major component of their diets.

The mirex residues showed a slight increase up to the November sample (Posttreatments 2 through 4) and dropped in the January sample (Posttreatment 5). Insect life was abundant in the area through November and dropped significantly afterward, corresponding to the trend of mirex residues in tissues. Collins et al. (*5*) reported a similar buildup of mirex in bobwhite quail in whole-body tissue; a peak occurred approximately 3 months after treatment.

A sharp rise occurred in residues in the Posttreatment 6 sample, which could be attributed to several factors. New plants and insects emerged in February 1976 due to unusually warm conditions and the residue peak may have reflected the quails' selection of these items for food. Barrier et al. (*2*) studying whitetail deer noticed a spring peak in DDT residues which they speculated to have been translocated from new spring growth consumed by the deer. Uptake and translocation of mirex by bean and pea seedlings have been reported by Mehendale et al. (*11*). Crop content examination of quail collected in Posttreatment 6 revealed new spring growth, seeds, and insects. Furthermore, fat reserves were low after the winter, possibly causing residue

concentration. Mirex probably entered quail through insect consumption; however, the variables listed above may be magnifying the situation. Residues were high in quail of Posttreatment 8 the summer after the 1975 pretreatment. This likely reflects mirex spraying of the study area rather than the spring 1975 treatment by SCPPRS of border areas. Here quail were exposed directly to mirex bait, rather than indirectly contacting mirex through the food chain.

In pen studies designed to test the toxicological effects of mirex in birds, Hyde et al. (*8*) found no reproductive inhibition in mallard ducks (*Anas platyrhynchos*) fed up to 100 ppm mirex, although some of their ducklings died. Analysis of selected duck tissues from the 100 ppm treatment indicated 2,964 ppm mirex in fat and 37 ppm in breast muscle. Heath et al. (*7*) determined the LC_{50} in 5-day diets of 2-week-old bobwhite quail to be 2,511 ppm mirex in feed. Pen studies testing the effects of 40 ppm mirex in the diet of bobwhite quail revealed no reproductive effects as measured by rates of egg production, shell cracking, embryonation rates, embryo survival, hatching, and hatchling success (*15*). Carcass residues average 113 ppm mirex (wet weight) in males and 172 ppm in females (*15*).

In a similar laboratory study of bobwhite quail receiving mirex treatments of 1, 20, and 40 ppm in feed, Kendall et al. found no harmful reproductive effects (1976: unpublished data). Birds analyzed after the 40 ppm treatment indicated 343 ppm and 197 ppm mirex (dry weight) in adipose tissue of males and females, respectively. Birds exposed to the 1 ppm treatment (F_0 generation) had 17 ppm mirex in fat deposits in males and 6 ppm in females. Furthermore, no reproductive inhibition was noted when a second generation of birds was exposed to 1 ppm mirex. The residue levels in tissue on the F_0 generation quail exposed to 1 ppm mirex closely correlates with residues presented in Table 2.

Action guidelines of the USDA set by the U.S. Environmental Protection Agency (EPA) for mirex residues in meat (lipid weight) of domestic animals intended for human consumption is 0.1 ppm (*4*).

Table 2 reveals that numerous breast muscle residues appear to be above EPA limits. In evaluating these EPA prescribed tolerance levels, Clark and McLane (*4*) reported that in woodcock (*Philohela minor*), breast muscle contains only 1.9 ± 0.1 percent fat, compared to hamburger which may contain up to 28 percent fat, or 15 times the mean percent fat of woodcock. Therefore, in considering the safety of chlorinated hydrocarbon residues in woodcock, it would be appropriate to multiply guideline levels by 15. Tolerance levels have not been established for mirex residues in bobwhite quail. However, in light of lipid weight ppm comparisons, the authors conclude that residue levels reported here would contaminate little if any of the human food chain. The mirex residue data in Table 2 would be further lowered if converted from the reported dry-weight to wet-weight concentrations; approximate conversion factors are fat, 0.77, and breast, 0.29.

The authors conclude that the current rate of mirex bait application (*12*) does not inhibit reproduction of the bobwhite quail. Furthermore, no significant human food chain contamination with mirex would be indicated in the consumption of quail harvested from a mirex-treated area.

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PCB's in Fish from Selected Waters of New York State [1]

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ABSTRACT

PCB residues in fish from 41 stations throughout New York State were monitored in 1975. Nearly all fish contained PCB's in detectable amounts although the levels of contamination and specific Aroclor varied. The Hudson River contained the highest known PCB concentrations within the United States; levels often exceeded 100 ppm. Other waters and fish which were significantly contaminated include Lake Ontario salmonids and Cayuga Lake lake trout. Onondaga Lake, previously closed to fishing because of mercury contamination, also appears to have abnormally high levels of PCB's approaching in some instances the action level of the Food and Drug Administration, U.S. Department of Health, Education, and Welfare. Samples from marine waters generally have contaminant levels substantially below 5.0 ppm.

A summary of PCB data collected during 1970–75 is presented.

Introduction

The Division of Fish and Wildlife, New York State Department of Environmental Conservation, began analyzing fish flesh for DDT in the early 1960's. This work was conducted in response to data suggesting that chlorinated hydrocarbons may interfere with reproduction of fish and wildlife resources. Results of the investigation led to State restrictions on use of DDT in 1965. During this time, research indicated that DDT analyses could be biased through sample contamination with similar compounds, polychlorinated biphenyls (PCB's) (*8*). To minimize errors in future work, both DDT and PCB's were extracted and analyzed to obtain separate values. From 1971 through 1975 PCB's were evaluated in all fish flesh analyses as a part of DDT monitoring.

The recent concern over the impact of PCB's on human health and their possible effect on New York's natural resources as well as reports of their ubiquitous nature (*6, 13, 20*) precipitated a shift from low-level monitoring to the statewide monitoring described in this report. The new program promulgated by the Department of Environmental Conservation in August 1975 revised all existing directions. Specific fish species and desired sizes were defined (Table 1). Stations were located in most large waterways throughout the State where PCB contamination could occur (Figure 1, Table 2) and, when possible, where earlier samples had been taken. Several Adirondack streams were included to ascertain statewide ambient conditions. Where contamination was known to exist sampling was intensified. This was the case of the Hudson River where inordinately high concentrations of the contaminant in fish were reported by the U.S. Environmental Protection Agency (EPA (*16*).

The objectives of this program were to determine the level and extent of PCB contamination in fish inhabiting or migrating through major waterways in New York State and to isolate contaminated portions or reaches of waterways, thereby identifying specific areas to be addressed by agencies which control water pollution.

TABLE 1. *Desired species and size distribution of fish from New York waters analyzed for PCB content, 1975*

Species	Small [1]	Medium [2]	Large [3]
	Length, in.		
Smallmouth bass	5–7	10–12	14–16
Largemouth bass	5–7	10–12	14–16
Walleye	7–9	11–13	19–21
Bullheads	4–6	8–10	11–13
Carp	5–7	13–15	23–25
Goldfish	4–6	7–9	10–12
Suckers	6–8	10–12	17–19
Striped bass	11–13	16–18	28–32
	Weight, lb. [4]		
Salmon (coho)	1–2	4–6	8–12
(chinook)	3–5	8–12	15–20
Rainbow or steelhead trout	1–2	3–5	5–8
Lake trout	1–2	4–6	8–12

[1] Minimum number of fish per sample was 6.
[2] Average number of fish per sample was 5.
[3] Maximum number of fish per sample was 5.
[4] Minimum number of fish per sample was 5.

[1] New York State Department of Environmental Conservation, Bureau of Environmental Protection, 50 Wolf Road, Albany, N.Y. 12233

FIGURE 1. *Fish collection stations for PCB studies, New York State–1975*

Stations selected for fish sampling were included in a major water column and sediment monitoring program conducted by the State Division of Pure Waters. Data from all sources are being used to track down sources of PCB contamination and to recommend remedial action.

Fish Collections

Regional Fish and Wildlife personnel were instructed on specific species and sizes of fish desired, general location of the stations, and timetable for collections. Sampling methods were primarily gill nets and electrofishing; angling and rotenone were used when other techniques were inadequate. On occasion, fish were donated by or purchased from commercial and recreational anglers.

The composition of the fish samples directed to the laboratories varied slightly, depending on the success of the collection teams. Specifications in Table 1 were flexible in order to accommodate a sampling program involved with diverse waters. Specific instructions requested that at each location members of two trophic levels, predators and forage species, be sampled in three size categories: small or sublegal, medium or minimally legal, and large.

The collection team chose the representative species and followed the length and number specifications in Table 1. In those areas where a commercial species is harvested, representative specimens were included.

All specimens were individually tagged, measured and weighed, placed by groups in polyethylene bags, and deposited in a cooler or freezer for shipment to a laboratory

TABLE 2. *Statewide sampling stations for PCB analysis, New York—1975*

Station No.[1]	Watershed	Waterway	Location
1	Upper Hudson River	Hudson River	Above Corinth
2			Above Glens Falls
3			At Fort Edward
			below General Electric Co.
4			5 miles below Fort Edward
5			Waterford area
6	Lower Hudson River		Coxackie-Catskill area
7			Lower Hudson
			Mouth of Rondout Creek
			Mouth of Wappingers Creek
			Mouth of Esopus Circle
			Kingston/Rhinecliff Bridge
			Tappan Zee Bridge area
			West Point
			George Washington Bridge area
			Highland/Poughkeepsie area
8		Rondout River	Rondout River above first dam
9	Mohawk River	Mohawk River	Below Schenectady
10			Below Oriskany
11	Seneca/Oneida/Oswego Rivers	Seneca/Oneida/Oswego Rivers	Below Fulton
12		Erie Canal	Below Clyde
13	Lake Erie/Niagara River	Niagara River	Below Lewiston
14	Lake Erie/Niagara River	Lake Erie	Dunkirk
15	Lake Ontario	Lake Ontario	Off Rochester
16			Oswego
17			Pulaski (salmonids)
18	Genesee River	Genesee	Below Industry
19	Chemung River	Chemung	Chemung
20	Susquehanna River	Susquehanna	Smithboro
21	Delaware River	Delaware	Below Pt. Jervis (State line)
22	Lake Champlain	Lake George	Bolton Landing
23		Lake Champlain	Ticonderoga
24			Plattsburg
25	St. Lawrence River	St. Lawrence	Cape Vincent
26			Massena
27	Black River	Black River	Watertown
28	St. Lawrence River	Raquette River	Norfolk
29		Fulton Fish Market	
30		Albany Fish Market	
31	Seneca/Oneida/Oswego Rivers	Onondaga Lake	
32	Upper Hudson River	Walloomsac/Hoosick Rivers	
33	Long Island	Marine Waters	
34	Upper Hudson River	Champlain Canal	Lock 8
35	Genesee River	Hemlock Lake	
36		Canadice Lake	
37	Seneca/Oneida/Oswego Rivers	Canandaigua Lake	
38		Keuka Lake	
39		Seneca Lake	
40		Cayuga Lake	
41		Seneca River	Montezuma National Wildlife Refuge

[1] See map, figure 1.

or storage center. Common names of fishes used throughout this report are those adopted by the American Fisheries Society (*1*) and are presented with their scientific nomenclature in Table 3.

TABLE 3. *Common and scientific names of fish and shellfish species analyzed for PCB's—1975*

Common Name	Scientific Name
Spiny dogfish	*Squalus acanthias*
Atlantic sturgeon	*Acipenser oxyrhynchus*
American eel	*Anguilla rostrata*
Blueblack herring	*Alosa aestivalis*
Alewife	*Alosa pseudoharengus*
American shad	*Alosa sapidissima*
Atlantic menhaden	*Brevoortia tyrannus*
Cisco or lake herring	*Coregonus artedii*
Coho salmon	*Oncorhynchus kisutch*
Chinook salmon	*Oncorhynchus tshawytscha*
Rainbow trout	*Salmo gairdneri*
Atlantic salmon	*Salmo salar*
Brown trout	*Salmo trutta*
Lake trout	*Salvelinus namaycush*
Rainbow smelt	*Osmerus mordax*
Chain pickerel	*Esox niger*
Northern pike	*Esox lucius*
Goldfish	*Carassius auratus*
Carp	*Cyprinus carpio*
White sucker	*Catostomus commersoni*
Silver redhorse	*Moxostoma anisurum*
Shorthead redhorse	*Moxostoma macrolepidotum*
Brown bullhead	*Ictalurus nebulosus*
Channel catfish	*Ictalurus punctatus*
Goosefish	*Lophius americanus*
Atlantic cod	*Gadus morhua*
Atlantic tomcod	*Microgadus tomcod*
Mummichog	*Fundulus heteroclitus*
Atlantic silverside	*Menidia menidia*
White perch	*Morone americana*
White bass	*Morone chrysops*
Striped bass	*Morone saxatilis*
Rock bass	*Ambloplites rupestris*
Redbreast sunfish	*Lepomis auritus*
Pumpkinseed	*Lepomis gibbosus*
Bluegill	*Lepomis macrochirus*
Smallmouth bass	*Micropterus dolomieui*
Largemouth bass	*Micropterus salmoides*
Black crappie	*Pomoxis nigromaculatus*
Yellow perch	*Perca flavescens*
Walleye	*Stizostedion vitreum*
Bluefish	*Pomatomus saltatrix*
Weakfish	*Cynoscion regalis*
Scup	*Stenotomus chrysops*
Tautog	*Tautoga onitis*
Freshwater drum	*Aplodinotus grunniens*
Atlantic mackerel	*Scomber scomberus*
Bluefin tuna	*Thunnus thynnus*
Grubby	*Myoxocephalus aenaeus*
Summer flounder	*Paralichthys dentatus*
Winter flounder	*Pseudopleuronectes americanus*
Northern puffer	*Sphoeroides maculatus*
Shellfish	
Blue claw crab	*Callinectes sapidus*
Northern lobster	*Hommarus americanus*
Hard clam	*Mercenaria mercenaria*
Surf clam	*Spisula solidissima*
American oyster	*Crassostrea virginica*
Common mussel	*Mytilus edulis*

Laboratory Analyses

Four State laboratories collaborated in the analysis of fish flesh: Department of Environmental Conservation Laboratories at Rome and Stony Brook, Department of Agriculture and Market Food Laboratory in Albany, and the Department of Health Griffin Laboratory in Guilderland. Limited analyses were conducted in the Buffalo, N.Y., laboratory of the Food and Drug Administration, U.S. Department of Health, Education, and Welfare. Specific analytical techniques have been described (*5, 9, 10, 14, 15, 17, 21*).

Frozen fish samples were defrosted, generally prepared as described below, ground three times, and blended.

FISH SIZE	PREPARATION
Very small: less than 100 g	Whole fish composites; see Table 4.
Small: 100–150 g	Head and viscera removed. Individuals composited.
Medium: 25–61 cm	Head and viscera removed. Composite if needed.
Large: 61–76 cm	Left fillet or anterior half of left fillet with skin intact. Composite if needed.
Jumbo: over 76 cm	Anterior third of left fillet with skin intact.

Brown bullhead, American eel, and Atlantic Ocean fish were the only species skinned before grinding; this is the common method of preparing them for consumption. Composites of medium through jumbo fish were made of ground and blended flesh; total weight of each individual was proportional to the total weight of the composite.

Laboratories performing analyses are noted in Tables 4 and 5. Recoveries by the analytical techniques cited above are 85 percent or more. Data were not adjusted to reflect recovery rate. Each procedure has a sensitivity of 0.01 ppm although two laboratories reported results only as low as 0.1 ppm. Very limited numbers of fish from Lake Ontario were subjected to mass spectrometry for confirmation of analytical results.

A quality assurance program was implemented to assure that results from the various laboratories were comparable. Table 6 shows that none of the participating laboratories had contamination problems; all reported zero for sample A. At the level of approximately 1 μg/ml of extract, which corresponds to the action level of 5 μg/g in fish, precision among the laboratories was remarkably good. No gross bias existed, although the laboratories had standard Aroclor mixtures obtained at different times and one laboratory used Aroclor 1242 for quantitation instead of Aroclor 1016. Results at higher concentrations indicate similar agreement.

In Table 6, the two sets of results from Griffin Laboratory illustrate the effects of using separate standards to quantitate the two Aroclor types and of using a mixed standard. Although all laboratories used individual standards, Table 6 results indicate basic similarity to results obtained for a mixed standard, Aroclors 1242 and 1254. This is probably due to differing gas chromatograph column conditions, and differing techniques of measuring the various Aroclors. To measure check samples such as these the use of individual Aroclors is the most desirable, but for environmental samples which are nearly always mixtures, use of mixed standards is analytically faster and probably more accurate. Table 7 supports this contention; some laboratories used mixed standards and some used indi-

TABLE 4. *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2]		
							Weighted Average	High	Low
Hudson River	1, 2	DEC–R	Smallmouth bass	1242/1016	26	12	0	0	–
				1254			TR	TR	0
			Walleye	1242/1016	2	2	0	0	–
				1254			TR	TR	0
			Yellow perch	1242/1016	15	11	0	0	–
				1254			TR	TR	0
			White sucker	1242/1016	4	4	0	0	–
				1254			TR	TR	0
Hudson River (Fort Edward below General Electric Company)	3	DEC–R	Walleye						
			Whole fish	1242/1016	14	2	91.01	104.21	81.12
				1254			36.01	41.80	28.29
			Edible flesh	1242/1016	4	1	9.88	9.88	–
				1254			8.31	8.31	–
			Yellow perch						
			Whole fish	1242/1016	30	2	164.36	236.40	128.33
				1254			37.14	62.88	24.27
			Edible flesh	1242/1016	10	1	61.10	61.10	–
				1254			18.24	18.24	–
			Rock bass	1242/1016	2	1	27.35	27.35	–
				1254			ND		
			Alewife	1242/1016	1	1	20.35	20.35	–
				1254			ND		
			White sucker	1242/1016	9	2	41.74	82.88	8.83
				1254			23.10	47.57	2.72
Hudson River (5 miles below Fort Edward)	4	DEC–R	Smallmouth bass	1242/1016	1	1	100.27	100.27	–
				1254			22.64	22.64	–
			Walleye	1242/1016	3	3	56.73	157.27	5.19
				1254			10.67	20.74	5.56
			Yellow perch						
			Whole fish	1242/1016	26	2	87.29	88.18	34.31
				1254			30.69	34.21	18.95
			Edible flesh	1242/1016	3	1	35.54	35.54	–
				1254			15.01	15.01	–
			White sucker [3]	1242/1016	10	5	47.30	78.00	28.03
				1254			19.82	41.10	8.13
			Brown bullhead	1242/1016	5	2	77.04	85.30	73.24
				1254			37.23	38.52	36.38
Hudson River (Stillwater)	4A	DEC–R	Largemouth bass						
			Whole fish	1242/1016	8	2	11.65	14.31	8.99
				1254			8.42	9.84	6.99
			Edible flesh	1242/1016	4	1	19.25	24.85	12.40
				1254			10.59	15.97	4.44
			American eel [4]	1242/1016	2	2	207.33	403.38	11.28
				1254			83.47	155.87	11.07
			Brown bullhead	1242/1016	1	1	4.66	4.66	–
				1254			7.99	7.99	–
			White sucker	1242/1016	6	2	58.32	154.28	10.34
				1254			15.93	35.92	5.94
Hudson River (Waterford)	5	DAM	Smallmouth bass	1242/1016	10	2	30.60	36.0	27.0
				1254			15.70	17.5	14.5
			Walleye	1242/1016	2	1	23.30	23.3	–
				1254			9.10	9.1	–
			White sucker	1242/1016	11	2	27.44	36.0	20.3
				1254			10.15	12.9	7.9
Hudson River (Below Troy Dam)	6, 7a-h	DEC–R	Striped bass						
			Whole fish	1242/1016 [5]	21	2	0.10	2.19	0
				1254			1.75	1.96	1.66
				1242/1016	42	31	12.05	56.42	0
			Edible flesh	1254	48	37	11.41	50.06	1.66

(Continued next page)

TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2] Weighted Average	High	Low
			Smallmouth bass	1242/1016	2	2	8.05	13.76	2.34
				1254 [6]	1	1	2.18	2.18	–
			Largemouth bass	1242/1016	10	10	5.43	19.73	0
				1254			4.62	7.65	1.73
			Yellow perch	1242/1016	7	2	1.19	4.18	0
				1254 [7]	5	1	5.28	5.28	–
			White perch	1242/1016	30	9	9.00	30.67	0
				1254 [8]	18	4	6.98	9.65	4.14
			Chain pickerel	1242/1016	1	1	8.68	8.68	–
				1254			ND		
Hudson River (Below Troy Dam)	6, 7a-h	DEC–R	Atlantic tomcod Whole fish	1242/1016	47	2	6.49	6.68	6.27
				1254			7.75	9.85	5.37
			Edible flesh	1242/1016	6	2	1.50	2.07	0.93
				1254			1.32	1.63	1.00
			American shad	1242/1016	15	6	1.35	9.00	0
				1254			1.14	1.88	0
			Blueback herring Whole fish	1242/1016	4	4	0.72	1.85	0
				1254			1.41	4.69	0
			Edible flesh	1242/1016	2	1	0	0	–
				1254			5.13	5.13	–
			American eel	1242/1016	11	7	54.34(25.61) [9]	186.22	0
				1254			27.20(19.15)	57.20	3.00
			Atlantic sturgeon	1242/1016	20	5	7.69	13.48	0
				1254			8.15	9.84	6.25
			Brown bullhead	1242/1016	1	1	0	0	–
				1254			3.58	3.58	–
			Atlantic menhaden	1242/1016	8	2	0	0	–
				1254			1.67	1.83	1.41
Rondout Creek	8	DAM	Largemouth bass	1242/1016	7	2	0	0	–
				1254			1.39	1.97	1.16
			White sucker	1242/1016	5	1	0	0	–
				1254			1.60	1.60	–
			Carp	1242/1016	3	1	0	0	–
				1254			1.50	1.50	–
Mohawk River (Schenectady)	9	DAM	Smallmouth bass	1242/1016	3	1	0.70	0.7	–
				1254			1.50	1.5	–
			White sucker	1242/1016	5	1	0.80	0.8	–
				1254			1.90	1.9	–
			Black crappie	1242/1016	9	2	1.11	1.6	0.5
				1254			1.13	1.4	0.8
			Largemouth bass	1242/1016	8	2	0.95	1.2	0.7
				1254			3.40	4.7	2.1
			Blueback herring	1242/1016	6	1	2.10	2.1	–
				1254			1.60	1.6	–
Mohawk River (Little Falls)	10	DAM	Walleye	1242/1016	10	2	4.45	4.9	4.0
				1254			9.25	9.5	9.0
			Smallmouth bass	1242/1016	2	1	3.90	3.9	–
							9.50	9.5	–
Mohawk River (Little Falls)	10	DAM	White sucker	1242/1016	11	2	0	0	–
				1254			2.79	4.2	1.1
			Brown bullhead	1242/1016	5	1	2.70	2.7	–
				1254			11.30	11.3	–
Seneca-Oswego Canal	11	DH	Smallmouth bass	1242/1016	14	3	2.29	4.4	0
				1254			1.13	2.5	0.7

(Continued next page)

TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2]		
							Weighted Average	High	Low
			Carp	1242/1016	10	2	2.00	4.0	0
				1254			5.60	7.2	4.0
Erie Canal	12	DH	Largemouth bass	1242/1016	1	1	0	0	–
				1254			0.80	0.8	–
			Northern pike	1242/1016	1	1	2.00	2.0	–
				1254			1.30	1.3	–
			Carp	1242/1016	8	2	0.60	1.6	0
				1254			0.30	0.8	0
Niagara River	13	DEC–R	Goldfish	1242/1016	5	1	0	0	–
				1254			TR	TR	–
			Largemouth bass	1242/1016	1	1	0	0	–
				1254			3.10	3.10	–
			Smallmouth bass	1242/1016	1	1	0	0	–
				1254			3.30	3.30	–
			White bass	1242/1016	1	1	0	0	–
				1254			11.31	11.31	–
Lake Erie	14	DEC–R/DAM	Smallmouth bass	1242/1016	19	4	0.19	0.4	0
				1254			0.72	1.0	0.6
			Walleye	1242/1016	19	9	0.19	0.6	0
				1254			0.70	1.45	TR
			Yellow perch	1242/1016	10	2	0.40	0.4	0.4
				1254			0.60	0.6	0.6
			Goldfish	1242/1016	4	1	1.00	1.0	–
				1254			1.40	1.4	–
			Brown trout	1242/1016	20	9	0.23	2.4	0
				1254			1.60	3.23	0.57
Lake Erie	14	DEC–R	Rainbow trout	1242/1016	10	9	0	0	–
				1254			2.03	3.77	0.76
			Chinook salmon	1242/1016	8	8	0	0	–
				1254			1.71	2.71	0.98
			Coho salmon	1242/1016	15	4	0	0	–
				1254			2.36	5.06	0.68
Lake Ontario	15–16	DEC–R	Coho salmon	1242/1016	23	15	0	0	–
				1254			8.05	18.65	2.09
			Chinook salmon	1242/1016	29	17	0.21	6.07	0
				1254			14.48	24.57	4.62
			Rainbow trout	1242/1016	9	9	0	0	–
				1254			5.76	12.35	1.47
			Brown trout	1242/1016	12	12	0	0	–
				1254			5.25	16.76	0.96
			Lake trout	1242/1016	19	14	0	0	–
				1254			9.38	14.62	1.35
			White perch	1242/1016	27	5	0	0	
				1248			0.05	0.48	–
				1254			4.12	12.06	2.14
			Yellow perch	1242/1016	25	4	0.38	0	
				1254			0.38	0.69	–
			Northern pike	1242/1016	17	10	0	0	–
				1254			1.79	4.02	0.96
			Largemouth bass	1242/1016	5	5	0	0	–
				1254			1.79	4.25	0.42
			Smallmouth bass	1242/1016	7	2	0	0	–
				1254			1.81	2.69	1.15
			Rock bass	1242/1016	11	2	0	0	0
				1254			0.83	3.58	0.55
			Brown bullhead	1242/1016	10	2	0	0	–
				1254			0.57	0.75	0.39

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TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2]		
							Weighted Average	High	Low
			Pumpkinseed	1242/1016	19	4	0	0	–
				1254			0.01	0.26	0
			Walleye	1242/1016	8	4	0	0	–
				1254			2.87	7.39	0.33
			Rainbow smelt	1242/1016	10	4	0	0	–
				1254			2.10	4.55	1.16
			American eel	1242/1016	2	2	0	0	–
				1254			6.28	10.74	1.82
			Black crappie	1242/1016	8	6	0	0	–
				1254			1.35	2.07	0.20
			White sucker	1242/1016	5	5	0	0	–
				1254			3.39	7.90	1.38
Lake Ontario (Salmon River/ Pulaski Weir)	17	DEC-R	Coho salmon	1242/1016	9	20	0	0	–
				1254			9.31	12.67	4.48
			Chinook salmon	1242/1016	21	15	0.11	2.28	0
				1254			6.98	17.01	2.84
Genesee River	18	DH	Smallmouth bass	1242/1016	13	3	1.77	4.0	0.5
				1254			2.23	5.6	0.5
			Silver redhorse	1242/1016	14	3	1.55	3.0	1.0
				1254			1.45	2.4	0.5
Chemung River	19	DAM	Smallmouth bass	1242/1016	12	3	0	0	–
				1254			0.88	1.5	0.6
			Rock bass	1242/1016	2	1	0.40	0.4	–
				1254			0.40	0.4	–
			White sucker	1242/1016	13	3	0.29	0.5	0
				1254			0.55	0.9	0.2
Susquehanna River	20	DH	Smallmouth bass	1242/1016	12	2	0.20	0.2	0.2
				1254			0.45	0.5	0.4
			Walleye	1242/1016	6	2	0.13	0.3	0.1
				1254			0.75	0.7	1.0
			White sucker	1242/1016	18	3	0.20	0.2	0.2
				1254			0.80	0.8	0.8
			Carp	1242/1016	11	2	0.41	0.5	0.3
				1254			1.25	1.3	1.2
Delaware River	21	DH	Smallmouth bass	1242/1016	3	2	1.33	1.6	0.8
				1254			0.67	0.8	0.6
			White sucker	1242/1016	5	1	0.40	0.4	–
				1254			0.50	0.5	–
			American eel	1242/1016	9	3	1.24	2.8	0
				1254			0.36	0.8	0
Lake George	22	DH & DEC-R	Lake trout	1242/1016	10	5	0.71	2.0	0
				1254			3.94	15.0	0
			Rainbow trout	1242/1016	6	6	0	0	–
				1254			<1.00	<1.00	<1.00
			Smallmouth bass	1242/1016	14	5	0.29	1.0	0
				1254			0.27	1.0	0
			Yellow perch	1242/1016	15	3	0.57	1.2	0
				1254			0.31	0.8	0
			Cisco	1242/1016	10	2	0.50	0.6	0.4
				1254			3.95	4.7	3.2
Lake Champlain	23, 24	DH	Smallmouth bass	1242/1016	29	6	0.75	3.0	0
				1254			1.21	3.5	0
			Walleye	1242/1016	5	1	0.30	0.3	–
				1254			0.20	0.2	–
			Northern Pike	1242/1016	4	2	0.2	0.3	0.1
				1254			0.4	0.6	0.2

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TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2]		
							Weighted Average	High	Low
			Yellow perch	1242/1016	17	3	0.40	0.4	0
				1254			0.59	0.8	0.2
			Brown bullhead	1242/1016	14	3	0.34	0.7	0
				1254			0.45	1.2	0
St. Lawrence River	25, 26	DEC-R	Smallmouth bass	1242/1016	16	9	0	0	–
				1254			4.58	6.85	1.62
			Walleye	1242/1016	11	3	0	0	–
				1254			1.83	2.05	1.58
Black River	27	DH	Walleye	1242/1016	8	2	1.25	2.0	0
				1254			3.11	4.5	0.8
			Brown bullhead	1242/1016	10	2	0.95	1.5	0.4
				1254			2.50	4.0	1.0
Raquette River	28	DH	Smallmouth bass	1242/1016	15	4	0.30	0.4	0.2
				1254			0.90	1.7	0.3
			Shorthead redhorse	1242/1016	14	3	0.32	0.4	0.2
				1254			0.44	0.6	0.3
Onondaga Lake	31	DEC-R	Smallmouth bass	1242/1016	9	3	1.82	1.9	1.2
				1254			3.13	4.0	1.2
			Walleye	1242/1016	1	1	3.10	3.1	–
				1254			2.50	2.5	–
			White perch	1242/1016	11	2	2.28	4.3	0.6
				1254			2.33	4.9	0.2
			White sucker	1242/1016	3	1	0.70	0.7	–
				1254			0.20	0.2	–
			Carp	1242/1016	7	2	2.20	3.0	0.2
				1254			6.04	7.5	2.4
			Brown bullhead	1242/1016	5	1	3.10	3.1	–
				1254			5.30	5.3	–
Walloomsack/Hoosic River	32	DH	Largemouth bass	1242/1016	5	2	2.62	2.8	2.5
				1254			2.98	3.5	2.2
			Yellow perch	1242/1016	4	1	2.00	2.0	0
				1254			3.40	3.4	0
			White sucker	1242/1016	7	3	1.73	2.9	0.7
				1254			1.58	2.9	0.6
Atlantic Ocean	33	DEC-SB	Bluefish	1242/1016	26	15	0	0	–
				1248			0	0	–
				1254			1.43	8.39	0.21
			Striped bass	1242/1016	29	17	0	0	–
				1248			0	0	–
				1254			1.05	3.59	TR
			Porgy	1242/1016	12	2	0	0	–
				1248			0	0	–
				1254			0.29	0.32	0.25
			Atlantic menhaden	1242/1016	18	7	0	0	–
				1248			0	0	–
				1254			1.07	1.53	0.44
			Spiny dogfish	1242/1016	60	4	0	0	–
				1248			0	0	–
				1254			0.25	0.27	0.22
			Bluefin tuna	1242/1016	7	7	0	0	–
				1248			0	0	–
				1254			1.42	6.68	0.19
			Atlantic mackerel	1242/1016+	5	5	0.46	0.52	0.31
				1254					
			Weakfish	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			0.21	0.21	–

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TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2] Weighted Average	High	Low
			Summer flounder	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			0.56	0.56	–
			Winter flounder	1242/1016	16	10	0	0	–
				1248			0	0	–
				1254			0.09	0.36	TR
			Atlantic cod	1242/1016	9	9	0	0	–
				1248			0	0	–
				1254			0.18	0.34	TR
			Tautog	1242/1016	3	3	0	0	–
				1248			0	0	–
				1254			TR	TR	TR
			Scup	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			0.33	0.33	–
			White perch	1242/1016	2	2	0	0	–
				1248			0	0	–
				1254			3.11	5.19	1.02
			Atlantic silversides Whole fish	1242/1016	50	2	0	0	–
				1248			0	0	–
				1254			0.98	1.51	0.45
			American eel	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			3.75	3.75	–
			Goosefish	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			TR	TR	–
			Northern puffer	1242/1016	1	1	0	0	–
				1248			0	0	–
				1254			TR	TR	–
			Hard clam	1242/1016	36	3	0	0	–
				1248			0.23	0.36	0.01
				1254			0	0	–
			Surf clam	1242/1016	6	1	0	0	–
				1248			0.36	0.36	–
				1254			0	0	–
			Blue claw crab	1242/1016	14	2	0	0	–
				1248			0.32	0.44	TR
				1254			0	0	–
			Northern lobster	1242/1016	1	1	0	0	–
				1248			0.13	0.13	–
				1254			0	0	–
			American oyster	1242/1016	48	4	0	0	–
				1248			0	0	–
				1254			TR	0.15	TR
			Common mussel	1242/1016	36	3	0	0	–
				1248			0	0	–
				1254			TR	TR	TR
Champlain Canal	34	DEC–R	Rock bass Whole fish	1242/1016	1	1	4.40	4.40	–
				1254			7.78	7.78	–
			Freshwater drum Whole fish	1242/1016	2	1	0	0	–
				1254			1.36	2.33	0.38
			Edible fish	1242/1016	2	2	0	0	–
				1254			TR	TR	–
			Yellow perch Whole fish	1242/1016	4	1	0	0	–
				1254			0.92	0.92	–
			Alewife	1242/1016	100	1	0	0	–
				1254			1.60	1.60	–

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TABLE 4 (cont'd.). *Concentrations of PCB's in fish and shellfish in New York State, 1975*

Water	Station	Laboratory [1]	Species	Aroclor	No. Fish Analyzed	Number of Analyses	Concentration of PCB's, ppm wet weight [2] Weighted Average	High	Low
			White sucker Whole fish	1242/1016	5	3	0	0	–
				1254			0.59	0.92	TR
			Pumpkinseed Whole fish	1242/1016	5	1	0	0	–
				1254			2.00	2.00	–
Hemlock Lake	35	DEC–R	Lake trout	1242/1016	10	10	TR	TR	0
				1254			TR	TR	0
			Rainbow trout	1242/1016	2	2	0	0	–
				1254			0	0	–
			Chain pickerel	1242/1016	9	5	0	0	–
				1254			0	0	–
			White sucker	1242/1016	3	1	TR	TR	–
				1254			0	0	–
			Carp	1242/1016	4	1	0	0	–
				1254			0	0	–
			Cisco	1242/1016	3	1	TR	TR	–
				1254			0	0	–
			Alewife	1242/1016	5	1	0	0	–
				1254			0	0	–
			Rainbow smelt	1242/1016	5	1	TR	TR	–
				1254			0	0	–
			Yellow perch	1242/1016	7	2	TR	TR	–
				1254			0	0	–
Canadice Lake	36	DEC–R	Lake trout	1242/1016	7	7	0	0	–
				1254					
			Rainbow trout	1242/1016	5	5	0	0	–
				1254					
Canandaigua Lake	37	DEC–R	Lake Trout	1242/1016	17	12	3.11	25.78	0
				1254			5.38	21.69	1.12
			Brown trout	1242/1016	1	1	0	0	–
				1254			2.58	2.58	–
Keuka Lake	38	DEC–R	Lake trout	1242/1016	18	8	TR	TR	0
				1254			0.09	1.77	0
			Rainbow smelt	1242/1016	6	1	0	0	–
				1254			0	0	–
			Alewife	1242/1016	9	2	0	0	–
				1254			0	0	–
Seneca Lake	39	DEC–R	Lake trout	1242/1016	10	10	0	0	–
				1254			6.42	15.99	2.62
Cayuga Lake	40	DEC–R	Lake trout	1242/1016	7	7	0	0	–
				1254			6.15	8.23	1.76
Montezuma National Wildlife Refuge	41	DEC–R	Carp	1242/1016	3	3	0.66	1.59	TR
							1.67	3.53	0.29

[1] Laboratory code: DEC–R = Department of Environmental Conservation, Rome Laboratory, New York State.
DAM = Department of Agriculture and Markets, Food Laboratory, New York State.
DH = Department of Health, Griffin Laboratory, New York State.
DEC–SB = Department of Environmental Conservation, Stony Brook Laboratory, New York State.

[2] – indicates absense of data, or no value less than 0 as reported in column titled "High."
TR = trace.
ND = not determined.

[3] Includes only fish analyzed for both aroclors; 4 fish not included.

[4] Sample includes one highly contaminated specimen.

[5] Sample does not include 6 fish for which Aroclor 1242/1016 was not determined.

[6] Sample does not include 1 fish for which Aroclor 1254 was not determined.

[7] Sample does not include 2 fish for which Aroclor 1254 was not determined.

[8] Sample does not include 12 fish for which Aroclor 1254 was not determined.

[9] Data in parentheses are weighted average PCB concentrations excluding two fish with exceptionally high concentrations.

TABLE 5. *Concentrations of PCB's in fish, 1970–74*

Water	Species	Year Collected	Aroclor	No. Fish Analyzed[1]	Number of Analyses	Concentration of PCB's, ppm wet weight		
						Weighted Average	High	Low
Hudson River (Warrensburg)	Smallmouth bass	1970	1254	1	1	0.18	0.18	–
Hudson River (Below Troy Dam)	Largemouth bass	1970–72	1254	20(19)	19(18)	6.58(4.09)	53.81	0.66
	Smallmouth bass	1970–72	1254	4(3)	4(3)	6.42(2.70)	17.58	2.56
	Striped bass	1970–72	1254	3	3	4.75	10.48	1.67
		1973	1254	22	22	14.75	49.63	3.70
	Atlantic sturgeon	1970–72	1254	4	4	6.71	7.03	5.73
	Northern pike	1972	1254	1	1	17.78	17.78	–
	Yellow perch	1972	1254	13	2	0.79	0.92	0.79
	White perch	1972	1254	39	31	4.79	15.81	0.38
	Black crappie	1970–72	1254	3	2	0.78	1.42	0.46
	Pumpkinseed	1972	1254	21	3	0.47	0.64	0.46
	Bluegill	1972	1254	4	1	0.52	0.52	–
	Redbreast sunfish	1972	1254	12	1	0.75	0.75	–
Rondout Creek	Smallmouth bass	1970	1254	2	2	14.23	17.58	10.88
	Largemouth bass	1970	1254	7	7	5.05	9.52	2.60
Rampo River	Largemouth bass	1971	1254	1	1	0.57	0.57	–
Lake Champlain	Smallmouth bass	1971	1242/1016	1	1	0.80	0.80	–
	Largemouth bass	1971	1242/1016	1	1	4.21	4.21	–
	Walleye	1971	1242/1016	2	2	28.15	55.46	0.84
	Northern pike	1971	1242/1016	1	1	0.80	0.80	–
	Chain pickerel	1971	1242/1016	1	1	0.15	0.15	–
	Atlantic salmon	1971	1242/1016	1	1	0.98	0.98	–
	Black crappie	1971	1242/1016	1	1	1.45	1.45	–
	Channel catfish	1971	1242/1016	2	2	7.00	7.85	6.15
	Brown bullhead	1971	1242/1016	1	1	0.68	0.68	–
	Freshwater drum	1971	1242/1016	1	1	0.70	0.70	–
	Carp	1971	1242/1016	1	1	1.94	1.94	–
	White sucker	1971	1242/1016	1	1	0.50	0.50	–
	Yellow perch	1971	1254	1	1	TR	TR	–
	White fish (species not known)	1971	1254	1	1	0.87	0.87	–
Lake Erie	Coho salmon	1973	1254	13	13	1.30	2.91	0.60
		1974	1254	26	26	1.74	5.88	TR
	Chinook salmon	1973	1254	4	4	1.48	1.89	1.20
		1974	1254	4	4	2.98	4.92	2.24
	Smallmouth bass	1971	1254	1	1	0.42	0.42	–
	Freshwater drum	1971	1242/1016	1	1	1.72	1.72	–
Niagara River	Smallmouth bass	1971	1242/1016	2	2	11.30	20.27	2.32
	Walleye	1971	1242/1016	1	1	1.02	1.02	–
	Freshwater drum	1971	1242/1016	2	2	16.54	23.57	9.51

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TABLE 5 (cont'd.). *Concentrations of PCB's in fish, 1970–74*

Water	Species	Year Collected	Aroclor	No. Fish Analyzed[1]	Number of Analyses	Concentration of PCB's, ppm wet weight		
						Weighted Average	High	Low
Lake Ontario	Coho salmon	1971	1254	9	9	6.67	21.97	0.66
		1974	1254	29	29	6.26	10.72	2.00
	Chinook salmon	1971	1254	12	12	21.45	34.48	9.24
		1974	1254	9	9	7.15	14.77	1.54
Lake Ontario	Rainbow trout	1974	1254	3	3	1.97	2.81	1.45
	Brown trout	1974	1254	4	4	7.69	14.50	2.70
	Northern pike	1970	1254	4	4	5.32	12.86	1.38
	Smallmouth bass	1970	1242/1016	3	3	7.11	8.81	4.38
			1254	2	2	3.88	4.52	3.24
	Largemouth bass	1970	1254	1	1	2.84	2.84	–
	Rock bass	1970	1254	2	2	0.49	0.59	0.40
	White bass	1970	1254	3	3	10.26	15.46	6.37
	White perch	1970	1254	3	3	3.42	5.15	2.12
	Carp	1970	1254	1	1	0.71	0.71	–
	Rainbow smelt							
	Whole	1972	1254	10	10	4.02	7.12	2.26
	Edible	1972	1254	11	11	2.16	3.42	1.15
Lake Ontario (Salmon River/ Pulaski Weir)	Coho salmon	1970	1254	17	17	7.92	12.10	5.01
		1971	1254	20	20	10.35	21.43	0.83
		1972	1254	20	20	4.65	13.29	0.82
		1974	1254	5	5	7.05	11.29	3.48
Lake Ontario (Salmon River/ Pulaski Weir)	Chinook salmon	1971	1242/1016	2	2	19.38	28.11	10.65
			1254	4	4	9.37	12.10	5.98
		1973	1254	51	51	5.81	13.14	1.45
	Rainbow trout	1971	1254	3	3	7.24	8.38	5.76
Lake Ontario (Little Salmon River)	Coho salmon	1971	1254	3	3	2.60	6.10	0.76
	Chinook salmon	1971	1254	4	4	15.75	29.36	4.68
Lake Ontario (Spring Brook)	Coho salmon	1972	1254	1	1	6.81	6.81	–
St. Lawrence River	Smallmouth bass	1971	1254	7	7	5.35	9.76	1.31
	Walleye	1971	1242/1016	1	1	3.42	3.42	–
	Sturgeon (species not known)	1971	1242/1016	1	1	11.89	11.89	–
	Channel catfish	1971	1242/1016	2	2	65.85	81.79	49.91
Allegheny River	Smallmouth bass	1971	1242/1016	2	2	2.21	2.36	2.06
	Muskellunge	1970	1242/1016	1	1	2.21	2.21	–
Genesee River-Lower portion	Smallmouth bass	1971	1254	2	2	7.36	10.59	4.12
	White bass	1971	1254	1	1	7.76	7.76	–
Chemung River	Smallmouth bass	1971	1254	3	3	0.46	0.62	0.23
	Walleye	1971	1254	2	2	1.02	1.09	0.94
	Yellow perch	1971	1254	2	2	0.14	0.28	TR
Cohocton River (above Bath)	Smallmouth bass	1971	1254	1	1	0.90	0.90	–
	Brown trout	1971	1254	1	1	2.80	2.80	–

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TABLE 5 (cont'd.). *Concentrations of PCB's in fish, 1970–74*

Water	Species	Year Collected	Aroclor	No. Fish Analyzed[1]	Number of Analyses	Concentration of PCB's, ppm wet weight		
						Weighted Average	High	Low
Mohawk River	Smallmouth bass	1971	1254	3	3	3.57	4.03	2.73
Barge Canal (Spencerport Holley)	Smallmouth bass	1971	1254	1	1	2.41	2.41	–
	Largemouth bass	1971	1254	1	1	1.48	1.48	–
Hoosic River	Smallmouth bass	1971	1254	2	2	2.49	2.62	2.35
	Largemouth bass	1971	1254	3	3	2.70	4.39	0.57
Raquette River	Smallmouth bass	1970	1254	1	1	0.32	0.32	–
	Yellow perch	1970	1254	1	1	0.27	0.27	–
	Pumpkinseed	1970	1254	1	1	0.81	0.81	–
Oswegatchie River	Smallmouth bass	1970	1254	7	7	1.51	4.85	0
	Largemouth bass	1970	1254	1	1	4.17	4.17	–
	Northern pike	1970	1254	5	5	0.9	0.46	0
	Yellow perch	1970	1254	9	9	0.12	1.07	0
	Pumpkinseed	1970	1254	1	1	TR	TR	–
	White sucker	1970	1254	1	1	20.92	20.92	–
Black Lake	Smallmouth bass	1971	1254	1	1	0.13	0.13	–
Brant Lake	Largemouth bass	1971	1254	1	1	TR	TR	–
Cannonsville Reservoir	Brown trout	1971	1254	6	6	0.25	1.65	0
Chautauqua Lake	Smallmouth bass	1971	1254	1	1	0.16	0.16	–
	White sucker	1971	1254	1	1	TR	TR	–
Conesus Lake	Largemouth bass	1971	1254	1	1	0.34	0.34	–
Keuka Lake	Largemouth bass	1971	1254	1	1	0.54	0.54	–
	Rainbow trout	1971	1254	1	1	1.33	1.33	–
Lake George	Lake trout	1971	1254	6	6	4.74	6.07	2.77
Onondaga Lake	Smallmouth bass	1972	1254	14	14	0.71	2.08	TR
	White perch	1972	1254	7	7	1.58	2.62	0.70
	Yellow perch	1972	1254	4	4	0.31	0.68	0
	Northern pike	1972	1254	1	1	1.75	1.75	–
	Black crappie	1972	1254	4	4	0.98	1.54	0.49
	Freshwater drum	1972	1254	4	4	1.17	1.78	0.37
	Brown bullhead	1972	1254	4	4	0.44	0.89	0.12
	Channel catfish	1972	1254	3	3	8.39	16.53	1.40
	American eel	1972	1254	2	2	5.44	6.97	3.91
Oneida Lake	Smallmouth bass	1971	1254	1	1	0.80	0.80	–
Pepacton Reservoir	Brown trout	1971	1254	7	7	0.74	2.62	TR
Saratoga Lake	Smallmouth bass	1971	1254	1	1	1.16	1.16	–
Schoharie Reservoir	Walleye	1971	1254	3	3	0.05	0.16	0
	Brown trout	1971	1254	3	3	0	0	–
Waneta Lake	Chain pickerel	1971	1254	3	3	TR	TR	TR
Whitney Point Reservoir	Largemouth bass	1971	1254	1	1	0	0	–
	Walleye	1971	1254	5	5	TR	TR	0

(Continued next page)

TABLE 5 (cont'd.). *Concentrations of PCB's in fish, 1970–74*

Water	Species	Year Collected	Aroclor	No. Fish Analyzed [1]	Number of Analyses	Concentration of PCB's, ppm wet weight: Weighted Average	High	Low
	Black crappie	1971	1254	3	3	TR	TR	0
Atlantic Ocean	Striped bass	1972	1254	5	2	0.57	0.59	0.57
	Bluefish	1972	1254	5	5	1.82	8.49	0.07
	Winter flounder	1973–74	1254	180	9	0.13	0.26	0
	Atlantic silverside	1973–74	1254	900	9	0.16	0.30	0
	Mummichog	1973	1254	11	1	0.30	0.30	–
	Grubby	1973	1254	20	1	0.19	0.19	–
	Spiny dogfish	1972	1254	4	4	0.25	0.27	0.21
	Northern lobster	1974	1254	1	1	0.15	0.15	–
	Oyster	1974	1254	36	3	0.34	0.75	0.12

NOTE: Results tabulated only for those fish for which the specific PCB was reported. All analyses conducted by Department of Environmental Conservation Laboratory, Rome, N.Y., except Atlantic Ocean analyses which were conducted by the Stony Brook Laboratory
– indicates absense of data.
TR = trace.
[1] Parenthetic numbers exclude one individual fish with high PCB concentration.

TABLE 6. *Laboratory comparisons of single aroclors in hexane and iso-octane solutions*

Sample Solution	Aroclor	Residues, µg/ml [1,2]: Known Standard	DAM	DEC–SB	DEC–R	DH	EPA
Hexane	1016	0	0	0	0	0	NS
	1242	0	0	0	0	0	NS
	1254	0	0	0	0	0	NS
Hexane	1016	0.80	0.89	1.09 [3]	1.15	0.71	NS
	1254	0	NV	NV	NV	NV	NS
Hexane	1016	0	NV	NV	NV	NV	NS
	1254	0.95	0.92	1.32	1.01	0.97	NS
Iso-octane (combined standards)	1016	55.5	73.3 [3]	NV	39.8 [4]	54.2(43.8)	26
	1254	72.0	92.8	NV	90.4	72.2(90.0)	91
	1016+1254	127.5	166.1	177 [3]	130.2	126.4(133.8)	117
Iso-octane (combined standards)	1016	61.0	70.0	NV	NV	NV (103.4)	38
	1242	64.5	50.0	NV	70.3	125 NV	60
	1016+1242	125.5	120.0	145 [4]			98

[1] Laboratory code: DAM = Department of Agriculture and Markets, Food Laboratory, New York State.
DEC–SB = Department of Environmental Conservation, Stony Brook Laboratory, New York State.
DEC–RL = Department of Environmental Conservation, Rome Laboratory, New York State.
DH = Department of Health, Griffin Laboratory, New York State.
EPA = U.S. Environmental Protection Agency Laboratory, Region II, New York City.
[2] NV = no value reported for this Aroclor.
NS = no sample sent to this laboratory.
[3] Residue reported as Aroclors 1242 and 1254.
[4] Residue reported as Aroclor 1242.

TABLE 7. *Laboratory comparison of PCB's in fish*

Sample ID	Aroclor	Residues, ppm [1, 2]					
		DAM	DEC–SB	DEC–R	DH	FDA	SD, % [3]
F4830	1016	NS	NS	NV	NS	2.32	
	1254			3.69		4.37	
F4831	1016	NV	NV	NV	4.3	NS	
	1254	3.3		2.58	6.0		45
	1242/1254		2.03 [4]				
F4842	1016	NV	NS	NV	1.0	4.34	
	1254	8.4		7.16	9.5	8.49	11
F4844	1016	NV	NV	NV	NS	NA	
	1254	6.6		5.55			
	1242/1254		2.63 [4]				
F4845	1016	NV	NV	NV	NS	NA	
	1254	4.2		6.34			
	1242/1254		2.23 [4]				

[1] Laboratory code: DAM = Department of Agriculture and Markets, Food Laboratory, New York State.
DEC–SB = Department of Environmental Conservation, Stony Brook Laboratory, New York State.
DEC–R = Department of Environmental Conservation, Rome Laboratory, New York State.
DH = Department of Health, Griffin Laboratory, New York State.
FDA = Food and Drug Administration Laboratory, New York, N.Y.

[2] NV = no value reported for this Aroclor.
NS = no sample sent to this laboratory.
NA = sample not analyzed.

[3] SD = standard deviation, determined when there were sufficient analyses for comparison.

[4] Reported as Aroclors 1242 and 1254.

vidual standards, but the agreement for Aroclor 1254 is reasonable. Unfortunately, no split samples contained much Aroclor 1016 but from the synthetic check samples there is no reason to believe that agreement would be different for this mixture.

Results

Tables 4 and 5 summarize results of the 1975 PCB analyses and compilation of earlier results. For the purpose of the discussion concentrations as a function of fish length are not discussed. Data are being compiled for use as a resource management tool wherein harvest of specific lengths may be regulated to protect the consumer while using the resource. Furthermore, concentration/length/species correlations may shift preferred species management in contaminated waters to favor that group of fish with a low propensity for PCB accumulation. Whole fish analysis results have been separated from flesh analysis results. The data are offered as a weighted average of each individual analyzed from a composite sample and are derived using the following formula:

$$\frac{({}^{X}n\,{}^{Y}n + {}^{X}n{+}1\,{}^{Y}n{+}1 + {}^{X}n{+}2\,{}^{Y}n{+}2 + \ldots)}{({}^{X}n + n{+}1 + {}^{X}n{+}2 + \ldots)} = \text{Weighted average (concentration of PCB's)}$$

Where, for each species of fish:

n, $n+1$, $n+2$, etc. = individual or group of fish analyzed.

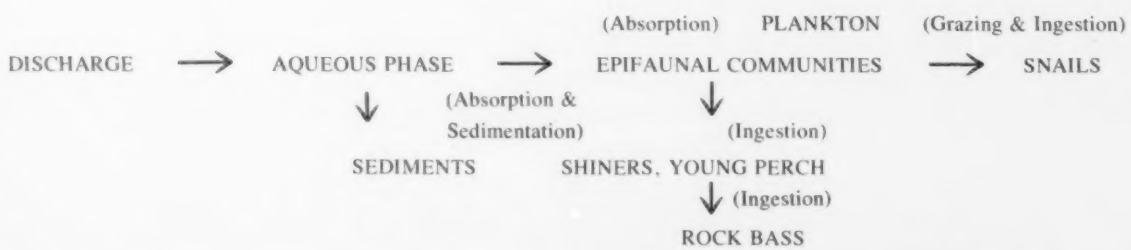
${}^{X}n$, ${}^{X}n+1$, ${}^{X}n+2$, etc. = number of fish in group n, $n+1$, $n+2$, etc.

${}^{Y}n$, ${}^{Y}n+1$, ${}^{Y}n+2$, etc. = concentration of PCB of group n, $n+1$, $n+2$, etc.

Zero concentrations were included in the weighted average. High and low individual readings in each sample are included to indicate end points of the data.

Discussion

PCB's are a ubiquitous contaminant of most environments. The source of this manufactured compound varies but appears to be primarily industries, open burning or incineration of solid wastes and waste oils, or migration from disposal sites (*13*). Once in the environment, PCB's, like organochlorine insecticides, are readily available for biomagnification since they are fat soluble compounds absorbed and stored in animal lipids. Nadeau (*16*) suggests a hypothetical transfer model for the biomagnification of PCB's in the Hudson River:



This model can be modified to show direct uptake by each biotic community from the aqueous phase (*11, 18, 19*). Through this avenue, a steady state may be achieved within a short period of time (±30 days) in highly contaminated waters (*23, 24*). Therefore, monitoring fish populations and analyzing properly composited samples for PCB contamination can indicate whether it is safe to consume fish flesh based on the Food and Drug Administration action level of 5 ppm, and to what degree a waterway is contaminated by PCB's. Indirectly, monitoring studies can show the need for specific studies to address the impact of PCB's on natural communities and/or populations, and the need to re-evaluate existing discharge permit limits.

The 1975 analyses confirmed that PCB's expressed as Aroclor 1016/1242 and/or 1254 were present in detectable amounts in most fish sampled. The level of contamination and specific Aroclor vary according to waterway size, location, and peripheral land use. Table 4 summarizes the data for edible flesh except in the cases indicated. Following is a general discussion of the findings in each waterway.

HUDSON RIVER; Stations 1–8, 32, 34. This major river system contains fish with the highest average concentration of total PCB's of any waterway sampled. Sampling nearly the entire extent of the river shows that contamination is widespread from Hudson Falls to the mouth of the river. The average level of contamination decreases in the lower reaches of the Hudson, perhaps through dilution from tributary waters, deposition in the sediments and aquatic organisms, and the effects of marine waters. Similar results occurred in fish collected from 1970 and 1973 (Table 5).

MOHAWK RIVER; Stations 9 and 10. Concentrations in 5 of 13 analyses in 1975 exceed the Food and Drug Administration action level of 5 ppm. Highest average concentrations appear upstream at the Little Falls station rather than at Schenectady, indicating a source of contamination above the town of Little Falls. The problem appears localized and does not affect the entire waterway as indicated by the Schenectady samples.

Earlier samples (Table 5) were either absent or too small to be compared.

SENECA/ONEIDA/OWSEGO RIVERS; Stations 11 and 12. PCB's are present, averaging 4 ppm. Concentrations are lowest at the Clyde station.

NIAGARA RIVER; Station 13. Largemouth and smallmouth bass have concentrations below 5 ppm; one white bass had levels above 10 ppm. The station was located below Lewiston, thus indicating no major source of PCB contamination from the Buffalo/Niagara Falls industrial complex. Earlier data are too sparse for direct comparison. However, additional study is warranted based on the 1971 analytical results and the single white bass noted above.

LAKE ERIE; Station 14. Samples from Lake Erie were taken at two locations, Dunkirk and the mouth of Cattaraugus Creek at Irving. The several species of fish contained levels of total PCB's well below 5 ppm. This is consistent with the 1973–74 data which indicated the presence of the contaminant at low concentrations.

LAKE ONTARIO; Stations 15–17. Generally, low levels of PCB's were found in warm water species, in contrast to the elevated concentrations commonly found in three species of salmonids taken from the lake proper. However, reduced levels were noted in the salmon that had migrated upstream in one major tributary, the Salmon River (Station 17). A reduction of the body fat through metabolic activities such as spawning and migration apparently caused the loss or displacement of some of the PCB burden in the edible flesh.

Comparison of the 1975 data with earlier findings is difficult. General trends indicate that concentrations similar to the present levels existed as early as 1970.

GENESEE RIVER; Station 18. Analysis of fish taken from the lower portion of the river below the village of Industry indicates a potential PCB problem in large fish. In two samples, one each of smallmouth bass and silver redhorse, levels were noted at or near 5 ppm. However, the 1971 samples, which were analyzed only for Aroclor 1254, indicate levels higher than those found in 1975. Due to the size of the earlier samples no trends can be identified except that PCB's (Aroclor 1254) are still present in substantial concentrations.

CHEMUNG/SUSQUEHANNA/DELAWARE RIVERS; Stations 19–21. These three independent river systems have been grouped for discussion since they reflect low levels of contamination, averaging 1 ppm or less. These figures are comparable to the samples collected in 1971 in the Chemung River.

LAKE GEORGE; Station 21. The larger, fatty lake trout of Lake George contain levels of PCB's (primarily Aroclor 1254) above 5.0 ppm. Lake George lake trout are known to have elevated levels of DDT (*4*). The analytical methodology used converted only DDT metabolites to dichlorobenzophenone rather than DDT itself. Therefore, PCB levels in lake trout and other species in Lake George, particularly ciscos, may be overstated. Further study of the lake is warranted.

LAKE CHAMPLAIN; Stations 23 and 24. These stations were located at Ticonderoga and Plattsburgh, respectively.

Reflected in the sampling are species in both the summer and winter fisheries as well as the limited commercial fishery. In all species sampled, total PCB's did not exceed 5 ppm although smallmouth bass from Plattsburgh approached that limit. These data are consistent with those obtained in 1971.

ST. LAWRENCE RIVER; Stations 25 and 26. Fish were collected from two stations, Massana (26) and Cape Vincent (25). Aroclor 1254 occurred but Aroclor 1242 did not. Walleye averaged about 1.8 ppm; smallmouth bass ranged from 1.62 to 6.85 ppm. Earlier data are consistent for the bass but suggest higher levels in lake sturgeon and catfish, species not sampled in 1975.

BLACK RIVER AND RAQUETTE RIVER; Stations 27 and 28. Fish from both the Black and Raquette Rivers had generally low levels of PCB contamination. These results were expected since the rivers are located in remote areas of the State away from industrial waters.

ONONDAGA LAKE; Station 31. Several species of fish, notably brown bullhead, white perch, smallmouth bass, and walleye, have concentrations of PCB's near or exceeding the FDA action level of 5 ppm. The lake has been closed to fishing since 1970 because of mercury pollution.

MARINE WATERS; Station 33. All species of fish and shellfish tested generally have total concentrations of PCB's less than 1 ppm. On rare occasions large oily fish such as bluefish have elevated levels; in one case the level exceeded 5 ppm.

CHAMPLAIN CANAL; Station 34. Fish from this waterway indicate the presence of PCB's, primarily Aroclor 1254, at low levels. A single individual rock bass, suspected to be a migrant from the Hudson River in the vicinity of Fort Edward, had levels above 5 ppm.

HEMLOCK, CANADICE, AND KEUKA LAKES, AND MONTEZUMA NATIONAL WILDLIFE REFUGE; Stations 35, 36, 38, and 41. These four waters have been grouped because they reflect low levels of PCB contamination.

CANANDAIGUA LAKE; Station 37. Chromatograms obtained for fish from this water were atypical; however, results are initially reported as PCB's. No use or spills of PCB's is known in the watershed.

SENECA AND CAYUGA LAKES; Stations 39 and 40. Previous use of small amounts of PCB's by local industry may have contaminated the fisheries. Results for Cayuga Lake (Station 40) generally confirm the findings of Bache et al. (*2*). Chromatograms for Seneca Lake display an atypical PCB pattern which is being investigated.

Conclusions

Nonindustrialized or lightly industrialized watersheds in New York State (Chemung, Susquehanna, Delaware, Black, and Raquette) contain fish with low levels of PCB's. Detectable amounts may result from atmospheric fallout, accidental spills, or land fill leachate. Several waterways have PCB's in amounts exceeding the action level of 5 ppm established by the Food and Drug Administration.

Rivers or waterways receiving contributions from industrialized watersheds contain organisms with high levels of PCB's. Hesse (*12*), Bremer et al. (*3*), and Veith (*22*) have indicated that levels in fish from the Great Lakes exceeded 5 ppm and that this level has not declined during the past five years. Information derived during the New York State 1975 study (Table 4) confirms these high concentrations in Lake Ontario. Large, long-lived, or fatty fish such as coho and chinook salmon, brown trout, and lake trout appear to bioaccumulate PCB's, generally above 5 ppm. A recent study (*2*) indicates that in salmonids the PCB level is directly and proportionally related to the age and oil of the fish. Findings in the present study generally support this premise.

The Hudson River below Hudson Falls contains fish with the highest level of total PCB's of any waterway sampled. Results above 50 ppm are not uncommon in the larger oilier fish; the highest individual concentration recorded was 559.25 ppm in a large eel. Comparison of the stations above Fort Edward with those immediately below indicates that a substantial PCB load is added to the river. This total loading probably does not emanate from atmospheric fallout or from other nondescript sources but rather from a major source of contamination or storage. Studies of water and sediment (*7*) have isolated industrial sources that are the subject of legal proceedings.

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RESIDUES IN SOIL

Organochlorine Insecticide Residues in Field Soils of the Kitakyushu District—Japan 1970-74[1]

M. Suzuki,[2] Y. Yamato,[2] and T. Watanabe[3]

ABSTRACT

Residues of organochlorine insecticides were monitored in field soils of the Kitakyushu District, Japan, from 1970 to 1974, before and after the insecticides were banned from agricultural fields. The arithmetic mean (AM) and geometric mean (GM) of BHC reached maximum levels in 1970 and 1971, then decreased rapidly during 1972 and 1974. Among BHC isomers, β-BHC had highest mean levels and was therefore thought to be the most persistent of the isomers in field soils. Residues of aldrin, dieldrin, and endrin peaked in 1970 and/or 1971, and then decreased yearly. The percentage of sites which had aldrin contamination have decreased; those with dieldrin contamination have increased. Mean values of p,p'-DDE, p,p'-DDT, and ∑DDT declined more slowly than did BHC and cyclodienes. Values of DDT and its related compounds were higher than were those of BHC, aldrin, dieldrin, and endrin. The residues of p,p'-DDE were most frequently detected of the DDT compounds.

Introduction

Insecticides are introduced into the soil environments by three major routes: direct incorporation of insecticides into field soils to kill soil-inhabiting pests, dislodging of insecticides from treated vegetable foliage, and wind drift during insecticide application.

Edwards (*5*) reported that the years required for 95 percent disappearance of applied insecticides (1–3 lb active ingredient/acre) in field soils was 1–6 (average 3 years) for aldrin, 3–5 (average 4 years) for chlordane, 4–30 (average 10 years) for DDT, 5–25 (average 8 years) for dieldrin, 3–5 (average 3.5 years) for heptachlor, and 3–10 (average 6.5 years) for lindane, respectively. Half-lives of applied insecticides have been reported by Nash and Woolson (*15*).

Insecticide residues have been detected in field soils 15 years after a single application of aldrin, DDT, and lindane (*10*), 15 years after BHC, DDT, and chlordane (*19*), and 20 years after an organochlorine pesticide application (*17*). Although many monitoring studies have been published (*3, 6, 8, 9, 14*), only limited information has been obtained since the ban of the organochlorine insecticides from agricultural fields.

In Japan, organochlorine insecticides such as BHC, DDT, aldrin, dieldrin, and endrin were applied extensively from the 1950's to the 1960's to control pests in rice paddies, vegetable fields, orchards, and forests. In late 1970, use of these insecticides was banned because of public concern with polluted foods. Among these insecticides, BHC had been applied the most extensively; the amounts of production in 1967 and 1968 were 41,742 tons and 45,695 tons, respectively. The ratio of BHC production to total arable land was approximately 0.81 lb/acre in 1968. That year, 767 tons of cyclodiene insecticides were imported, and 4,936 tons of DDT were produced. Since 1971, however, the production of these insecticides has almost ceased.

The present paper reports organochlorine insecticide residues and related compounds in field soils of the Kitakyushu District, Japan (Fig. 1), before and after a ban of these insecticides.

Sampling Procedures

Soil samples were taken from fields where the primary crops for many years had been vegetables such as radishes, cabbages, spinach, carrots, chinese cabbages, cucumbers, turnips, tomatoes, eggplants, and lettuce. Considerable amounts of organochlorine insecticides had been applied, although no detailed histories of insecticide applications could be obtained. The textures of the soil samples varied from loamy to sandy. Samples were collected in cores 15 cm deep and 10 cm in diameter at diagonal points of the field. Sampling schedules appear in Table 1.

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FIGURE 1. *Map of Japan showing Kitakyushu District*

Analytical Methods

EQUIPMENT AND REAGENTS

Kuderna-Danish evaporative concentrator with three-ball Snyder column with ground glass fittings
Chromatographic column, 20 by 300 mm
Five-ml graduated concentrator with a ground glass fitting
Squibb-type 1-liter separatory funnel
Buchner glass funnel, 25G4 (150-200 mesh)
Sanyo mixer, SM–1000
Shimadzu GC–5AIEE gas chromatograph with dual tritium-foil electron-capture detectors (300 mCi)
Acetonitrile redistilled in accord with AOAC methods (*1*)
n-Hexane redistilled twice in an all-glass distillation system
Reagent grade anhydrous sodium sulfate heated 2 hours at 625°C to eliminate contaminants
Redistilled water washed thoroughly with n-hexane
Insecticide standards with 99+percent purity

PRETREATMENT

The soil sample was dried at room temperature, pulverized with a mortar, and screened through a 20-mesh sieve. The screened soil was put into a ball mill jar, rotated without balls for 5 hours, and mixed well.

TABLE 1. *Schedule for soil sampling for organochlorine insecticide analysis, Kitakyushu District, Japan—1970—74*

Collection Dates	No. samples collected
November 26, 1970	24
July 13, 1971	19
September 14, 1971	4
November 5, 1971	45
July 16, 1972	17
November 17, 1972	20
July 9, 1973	16
December 13, 1973	22
November 21, 1974	33

EXTRACTION

All analyses were carried out in duplicate and the results represent an average of duplicate analyses. A 100–g sample was put into a mixer and 0.7 volume of distilled water per 100 g soil sample was added to deactivate. After 30 minutes, 200 ml of acetonitrile was added and the mix was blended 5 minutes at a high speed. An extract and the rinsings were filtered through a Buchner glass funnel, and the filtrate was poured into a 1–liter separatory funnel and shaken with 100 ml of n-hexane for 5 minutes. Then 600 ml of distilled water was added and the funnel was shaken for 1 minute to achieve partitioning. The resulting aqueous phase was drained and the n-hexane phase was washed twice with distilled water. The n-hexane phase was dried by passing through a column of 5 cm anhydrous sodium sulfate.

The dried n-hexane phase was concentrated by a Kuderna-Danish evaporative concentrator. Heptachlor epoxide was added as an internal standard and subjected into a gas chromatograph equipped with a dual channel electron-capture detector. Recovery was well above 90 percent; results were not corrected.

GAS CHROMATOGRAPHY

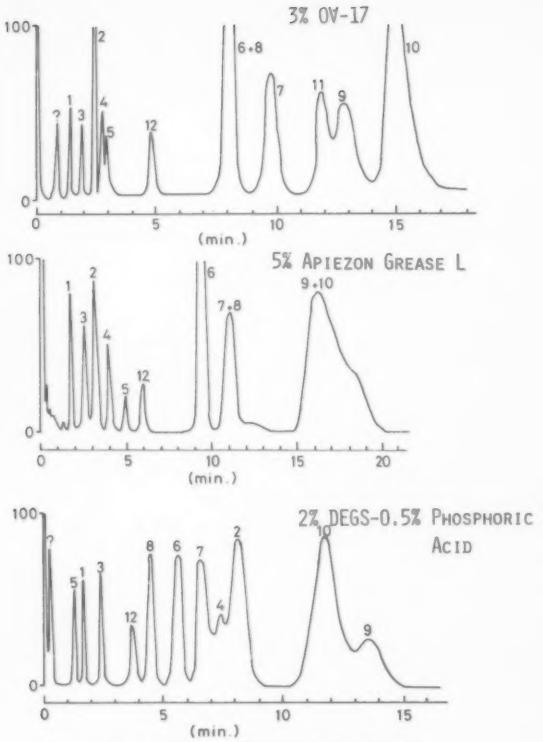
Organochlorine insecticides and related compounds in soil samples were identified and measured as described in the previous report (*22*).

Results and Discussion

The typical gas chromatograms of residual organochlorine insecticides and their related compounds in soil samples are shown in Figure 2. Results of the present monitoring study are presented in Table 2. For each year, the total sampling sites, the number and percent of sites which had insecticides and/or related compounds, the range of residue detected, and the arithmetic mean (AM) and geometric mean (GM) are given.

Residues of α–, β–, and δ–BHC were detected beside insecticidally active γ–BHC (lindane) in the soil samples analyzed, since crude BHC had been formulated and marketed as BHC insecticides in Japan. AM and GM values of BHC were highest during 1970 and 1971, then decreased yearly. The high residues in 1971, one year after the ban, reflect the application of stocked BHC purchased before the ban.

In 1970, the high value of 3.960 ppm reported for α–BHC occurred because the field had been sampled shortly after BHC application. Levels of BHC decreased considerably from 1971 to 1974. Soil in 1974 contained only 10.7 and 22.4 percent of the 1971 AM and GM mean levels of α–BHC, 21.6 and 18.4 percent of the levels for β–BHC, 16.3 and 25.4 percent for γ–BHC, and 17.3 and 16.0 percent for δ–BHC. These results indicate rapid dissipation of



1: α-BHC; 2: β-BHC; 3: γ-BHC; 4: δ-BHC; 5: aldrin; 6: dieldrin; 7: endrin; 8: p, p'-DDE; 9: p, p'-TDE; 10: p, p'-DDT; 11: o,p'-DDT; 12: heptachlor epoxide (internal standard).

FIGURE 2. *Typical gas chromatograms of insecticides and their related compounds in soils.*

BHC isomers from field soil. The average residue in 1970 was composed of 28 percent α–BHC, 44 percent β–BHC, 14 percent γ–BHC, and 14 percent δ–BHC. In 1974, ratios were 8 percent α–BHC, 77 percent β–BHC, 7 percent γ–BHC, and 8 percent δ–BHC. The marked increase in percentage of β–BHC in total BHC corroborates the previous report that β–BHC was the most persistent isomer of BHC in the soil (*21*); the residue levels of total BHC could be expected from the level of β–BHC. In contrast to β–BHC, α–BHC and γ–BHC might degrade considerably in field soil. All the BHC isomers were detected in all soil samples analyzed in 1970–73. In 1974, however, one sample had no γ–BHC and four samples had no δ–BHC, which illustrates the different degrees of persistence among BHC isomers in soil.

AM and GM values of aldrin and dieldrin peaked in 1971, then decreased yearly with minor exceptions. The detection ratios of aldrin were 8.3 percent in 1970, 26.8 percent in 1971, 48.6 percent in 1972, 26.3 percent in 1973, and 21.2 percent in 1974. Ratios were highest in 1972 and decreased gradually due to oxidative conversion and microbial degradation of aldrin to dieldrin in soil (*11, 12*). Detection ratios of dieldrin were 50 percent in 1970, 48.5 percent in 1971, 62.2 percent in 1972, 68.4 percent in 1973, and 75.8 percent in 1974. Unlike aldrin, dieldrin ratios increased yearly, due to the conversion of aldrin to dieldrin in soil. Although AM and GM values of dieldrin decreased yearly from 1970 to 1973, no marked decrease of these values was observed in 1974. The maximum AM and GM values of endrin were recorded in 1971, but considerable decreases occurred afterward. Values obtained in 1974 were about a tenth and a twelfth of those in 1971. For dieldrin, values were about a third and two-fifteenths of the 1971 values. Hence, of these stereo-isomers, the endo-type, endrin, was more degradable in soil than the exo-type, dieldrin. Nash et al. (*16*) reported that endrin was readily converted to its delta-ketone, alcohol, and aldehyde in soil with a pH of 6.4. This suggests that endrin is considerably degradable in the acidic soil common in arable fields of Japan.

AM and GM levels of DDT and ∑DDT were considerably higher than those of BHC and cyclodiene insecticides. Among DDT and related compounds, *p,p'*–DDT and *p,p'*–DDE were detected most frequently. Residues of *p,p'*–TDE and *o,p'*–DDT were detected in smaller quantities. Detection ratios of *p,p'*–DDE were 75.0 percent in 1970, 73.5 percent in 1971, 78.4 percent in 1972, and 100 percent in 1973 and 1974. Ratios increased yearly with minor exceptions due to the conversion of *p,p'*–DDT to *p,p'*–DDE in soil by dehydrochlorination. But detection ratios of *p,p'*–DDT have not decreased, suggesting widespread contamination of the soil by DDT as well as BHC. Cliath and Spencer (*4*) reported that the disappearance of DDT from well-aerated soil was due to the conversion to DDE and subsequent dissipation of DDE into the atmosphere. This suggests that DDT residing in field soil might degrade slowly to DDE; however, its dissipation might be accelerated later.

Insecticides in the environment can be degraded by physico-chemical action or by microbial action. Generally, the physico-chemical action occurs shortly after insecticide application, and microbial action occurs more slowly, among the persistent residues. In the soil, microorganisms are the most active factor in insecticide degradation. For instance, BHC is degradable in reductive conditions and in anaerobic microbe-rich soil (*13, 21*).

In the degradation process, γ–BHC is initially converted to γ–pentachlorocyclohexene, and secondly to γ–tetrachlorocyclohexene (*23*). Isomerization of γ–BHC to the other isomers was reported when γ–BHC was incubated with sediments from Pearl Harbor, Hawaii (*2*), and Lake Tomahawk, Wis. (*18*). DDT was easily converted to TDE when the soil was flooded and alfalfa was applied to the field as an organic matter (*7*). In this manner, insecticides might be dissipated from the soils; however, organochlorine insecticides studied here were generally far

TABLE 2. *Insecticide residues in soil from Kitakyushu District, Japan—1970–74*

Pesticide	No. Positive Sites	Percent Positive Sites	Residues, ppm dry weight		
			Range	Arith. Mean	Geom. Mean
		1970: 24 Sites			
α-BHC	24	100.0	0.006–3.960	0.233	0.0439
β-BHC	24	100.0	0.062–2.420	0.352	0.2158
γ-BHC	24	100.0	0.008–1.084	0.108	0.0456
δ-BHC	24	100.0	Tr –0.789	0.108	0.0551
∑BHC	24	100.0	0.076–8.253	0.791	0.4325
Aldrin	2	8.3	0.085–0.263	0.174	0.1496
Dieldrin	12	50.0	0.002–1.726	0.308	0.0769
Endrin	5	20.8	0.016–0.125	0.054	0.0275
p,p'-DDE	18	75.0	Tr –0.650	0.131	0.0543
p,p'-DDT	18	75.0	Tr –6.450	0.577	0.1820
∑DDT	18	75.0	Tr –8.254	0.810	0.2999
		1971: 68 Sites			
α-BHC	68	100.0	0.002–1.948	0.131	0.0425
β-BHC	68	100.0	0.021–2.612	0.631	0.3716
γ-BHC	68	100.0	0.003–0.965	0.080	0.0334
δ-BHC	68	100.0	Tr –0.748	0.081	0.0455
∑BHC	68	100.0	0.026–6.273	0.931	0.5210
Aldrin	25	36.8	0.002–1.012	0.074	0.0189
Dieldrin	33	48.5	0.024–1.014	0.294	0.1950
Endrin	21	30.9	0.036–0.654	0.191	0.1330
p,p'-DDE	50	73.5	0.005–2.048	0.167	0.0656
p,p'-DDT	40	58.8	0.015–9.801	1.259	0.5129
∑DDT	50	73.5	0.049–11.882	1.830	0.7481
		1972: 37 Sites			
α-BHC	37	100.0	0.002–0.800	0.044	0.0174
β-BHC	37	100.0	0.009–1.250	0.226	0.1492
γ-BHC	37	100.0	0.002–0.073	0.021	0.0178
δ-BHC	37	100.0	0.002–0.500	0.037	0.0189
∑BHC	37	100.0	0.026–6.273	0.328	0.2133
Aldrin	18	48.6	0.002–0.280	0.045	0.0220
Dieldrin	23	62.2	0.003–0.745	0.263	0.1089
Endrin	15	40.5	0.015–0.629	0.145	0.0847
p,p'-DDE	29	78.4	0.009–1.044	0.165	0.0867
p,p'-DDT	28	75.7	0.022–6.661	1.014	0.3467
∑DDT	29	78.4	0.031–10.994	1.487	0.5508

(Continued next page)

TABLE 2 (cont'd.). *Insecticide residues in soil from Kitakyushu District, Japan—1970–74*

Pesticide	No. Positive Sites	Percent Positive sites	Residues, ppm dry weight		
			Range	Arith. Mean	Geom. Mean
		1973: 38 Sites			
α-BHC	38	100.0	0.001–0.864	0.034	0.0071
β-BHC	38	100.0	0.005–0.686	0.121	0.0708
γ-BHC	38	100.0	Tr –0.399	0.025	0.0181
δ-BHC	38	100.0	Tr –0.125	0.018	0.0079
∑BHC	38	100.0	0.006–1.573	0.196	0.1069
Aldrin	10	26.3	Tr –0.007	0.002	0.0033
Dieldrin	26	68.4	0.002–0.250	0.063	0.0268
Endrin	19	50.0	0.006–0.109	0.038	0.0252
p,p'–DDE	38	100.0	0.001–0.831	0.117	0.0455
p,p'–DDT	38	100.0	0.006–4.174	0.686	0.2925
∑DDT	38	100.0	0.007–4.989	0.863	0.1808
		1974: 33 Sites			
α-BHC	33	100.0	0.001–0.067	0.014	0.0095
β-BHC	33	100.0	0.002–1.172	0.136	0.0682
γ-BHC	32	97.0	0.001–0.100	0.013	0.0085
δ-BHC	29	87.9	0.001–0.194	0.014	0.0073
∑BHC	33	100.0	0.003–1.533	0.177	0.1023
Aldrin	7	21.2	0.002–0.009	0.005	0.0039
Dieldrin	25	75.8	0.001–0.506	0.087	0.0263
Endrin	23	69.7	0.001–0.102	0.022	0.0109
p,p'–DDE	33	100.0	0.006–0.762	0.104	0.0388
p,p'–DDT	33	100.0	0.008–4.605	0.518	0.1049
∑DDT	33	100.0	0.018–5.504	0.671	0.2009

NOTE: Tr = trace (<0.001 ppm).
∑BHC = α-BHC + β-BHC + γ-BHC + δ-BHC.
DDT = *p,p'*–DDE + *p,p'*–TDE + *p,p'*–DDT + *o,p'*–DDT.

more persistent than are organophosphorus and/or carbamate insecticides.

Translocation of insecticides from field soil into vegetables has been reported (*12, 20*). In Japan, vegetables have been highly contaminated with organochlorine insecticides (*22*). Despite a recent ban on their use, residue levels often still exceed the pesticide residue tolerance set by the Japanese government. Therefore, organochlorine insecticides in field soil should be monitored during the next several years, in conjunction with the study of insecticide residues in vegetables.

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GENERAL

Dieldrin Residues in Soybeans in Illinois, 1965, 1966, 1967, 1971, and 1974 [1]

Steve Moore III, H. B. Petty, W. N. Bruce, Roscoe Randell, and D. E. Kuhlman

ABSTRACT

The Illinois soybean crop was monitored in 1965, 1966, 1967, 1971, and 1974 for dieldrin residues resulting from aldrin applied to corn in the years before soybean cultivation. Residue levels of dieldrin in soybeans increased between 1965 and 1974. The percent of fields which had soybeans with a dieldrin level above 0.03 ppm increased between 1965 and 1974. Dieldrin residue levels in soybeans grown in Illinois are expected to decline now that the use of aldrin has diminished and will soon cease. No significant correlation was evident between the dieldrin levels in soybeans and the area of the State where they had grown, the date of planting, or the variety.

Introduction

Aldrin has been the principal chlorinated hydrocarbon insecticide used on corn soil in the United States for the control of underground insects since the early 1950's. It was applied and incorporated into the soil at an average rate of 1.1 pounds of active ingredient per acre. According to Wedberg et al. (*13*), aldrin use reached its peak in Illinois in 1967 when 4,481,258 acres were treated (Table 1). Aldrin use has declined since that time due to the development of insect resistance and concern by farmers about residue problems. Decker et al. (*5*) showed that after an aldrin application small amounts of its epoxide, dieldrin, remained in the soil for at least 12 years and their disappearance could be predetermined with some certainty (Table 2). There is a rapid loss of adrin/dieldrin residues the first two years after the aldrin application due to the volatility of the aldrin and the desorption chracteristics of the soil. The determination was based on a 2-year half-life (continuous row cropping) and 4-year half-life (a rotation involving no-till cropping) after the first 2 years. Thus soil residues in a field can be calculated from the aldrin treatment history. The amount and persistence of residues vary with such factors as soil type, cropping history, weather, and cultivation.

Soybeans are exposed to dieldrin residues when growing in fields where aldrin has been used on corn in previous years. Rotation of corn and soybeans is a common practice in Illinois. From 1964 to 1974, 9 to 10 1/2 million acres of corn and 5 to 8 1/2 million acres of soybeans were grown annually. In 1959 and 1960, aldrin was tested for control-

TABLE 1. *Use of aldrin on corn soil in Illinois, 1956–1974* [1]

Year	Acres Treated with Aldrin	Year	Acres Treated with Aldrin
1956	314,679	1966	4,093,284
1957	558,677	1967	4,481,258
1958	663,326	1968	4,136,580
1959	1,126,417	1969	3,569,165
1960	1,607,689	1970	2,883,555
1961	2,187,740	1971	2,042,339
1962	2,979,354	1972	1,449,817
1963	3,441,920	1973	1,216,257
1964	3,407,908	1974	1,320,229
1965	3,635,546		

[1] Adapted from Wedberg et al., 1975, literature reference (*13*).

TABLE 2. *Theoretical residues (aldrin + dieldrin) to be expected in soil at yearly intervals after aldrin applications* [1]

Years after application	Two-year Half-Life	Four-year Half-Life
0	0.5	0.5
1	0.13	0.13
2	0.10	0.10
3	0.07	0.085
4	0.05	0.07
5	0.035	0.06
6	0.025	0.05
7	0.018	0.043
8	0.013	0.035
9	0.009	0.030
10	0.006	0.025

NOTE: Residues measured in ppm each spring after application of 1 lb./a. in top 6 inches of soil.

[1] Adapted from Decker, Bruce, and Bigger, 1965, literature reference (*5*).

[1] Illinois Cooperative Extension Service, Illinois Agricultural Experiment Station, University of Illinois, and Illinois Natural History Survey, 169 Natural Resources Building, Urbana, Ill. 61801.

ling soil-infesting insects in soybeans. An analysis of the soybeans showed the presence of aldrin/dieldrin residues. These chemicals are translocated from the soil to grain or fruit, as demonstrated by Lichtenstein (*7–10*), Bruce et al. (*2*), Bruce and Decker (*1*), and Eden and Arthur (*6*).

According to Bruce and Decker (*1*), the magnitude of the actual dieldrin residue in soybeans is about 6 to 11 percent (average 8 percent) of the actual dieldrin residue in the soil (Table 3). Turner et al. (*12*) showed that more dieldrin is present in the bean seeds from the lower half of the plant than in those from the upper half. Petty (*11*) reported the magnitude of dieldrin residues in soybeans in Illinois, and suggested that farmers allow at least 2 years to elapse between the last application of aldrin and the planting of soybeans in a field in order to produce soybeans free of significant dieldrin residues. The purpose of the present study was to determine the residue levels of dieldrin in soybean seeds produced in Illinois and to determine trends in dieldrin residue levels through the years.

Sampling Procedures

A random survey of Illinois soybeans was first conducted in 1965 to determine the levels of dieldrin residues (*3*). Similar surveys followed in 1966, 1967, 1971, and 1974. Using a system of random selection for objective yields, the Illinois Crop Reporting Service Enumerators obtained raw soybeans, and field histories of insecticide use and cropping practices. Between 52 and 106 fields of soybeans were randomly selected and sampled during each study year. Soybeans were collected in 1-quart screw-cap glass jars which were covered with aluminum foil before the cap was fastened. The samples were obtained by holding the jar at the edge of the stream of soybeans as they were augered or elevated into the bin or wagon. The samples were taken at harvest and submitted to the Illinois Natural History Survey for analysis. The analyses, which were based on the weight of the whole soybean, were accurate within ±0.001 ppm as shown in trial recoveries (Table 4).

TABLE 3. *Aldrin and dieldrin residues in soil, and corresponding dieldrin residue in soybeans grown in same soil after aldrin applicaton* [1]

Year	Residues, ppm		Percent Recovery
	Soil	Beans	
1961	0.390	0.043	11.0
1962	0.253	0.026	10.3
1963	0.174	0.017	9.8
1964	0.092	0.006	6.5

NOTE: Aldrin applied at 2 lb/a on April 14, 1961.

[1] Adapted from Bruce and Decker, 1966, literature reference (*1*).

TABLE 4. *Recovery of dieldrin added to soybean flour*

Dieldrin Added, ppm	Dieldrin recovered, ppm		
	Replicate 1	Replicate 2	Replicate 3
0.0000	0.0000	0.0040	0.0005
0.0010	0.0004	0.0008	0.0000
0.0020	0.0015	0.0017	0.0018
0.0050	0.0036	0.0051	0.0048
0.0100	0.0095	0.0110	0.0092
0.0200	0.0195	0.0172	0.0190

Analytical Technique

SAMPLES

Fifty-gram lots of soybeans were hexane-rinsed to remove harvest dust which may have contained trace amounts of dieldrin. Soybeans were dried and blended at high speed until reduced to a fine flour. This facilitated the complete extraction of oil from the 10–g flour aliquots used for each replicate. The soybeans contained about 21± percent oil by weight, but the analysis was based upon the weight of the whole soybean, and no special effort was made to survey oil contents of beans. Analyses were made in triplicate.

REAGENTS AND MATERIALS

All solvents used were nanograde or redistilled in glass. Florisil and Na_2SO_4 were baked at 450°C for 18 hours to remove moisture and impurities, then stored in glass-stoppered 1000–ml Erlenmeyer flasks.

EXTRACTION

Ten grams of soybean flour was added to a 250–ml Erlenmeyer flask with 150 ml 10 percent ether in hexane and heated overnight at 50°C. The mixture was filtered through No. 1 Whatman filter paper into another Erlenmeyer flask. The flour residue in the filter paper was rinsed several times with hexane to ensure complete transfer of the extracted oil. A few grains of Na_2SO_4 was added to the filtrate, a Snyder column was attached, and all but about 10 ml of the solvents were removed by distillation through the Snyder column.

PARTITIONING WITH ACETONITRILE

The extracted oil, approximately 2 g, was transferred to a 125–ml separatory funnel with four portions of 5 ml hexane. This hexane/oil mixture was extracted 4 times with 25–ml portions of acetonitrile which had been saturated with hexane. Each successive partition was shaken 100 times, the phases were allowed to separate, and the lower acetonitrile portion was drained into a 1000–ml separatory funnel.

Five hundred ml water and 100 ml hexane were added to the combined acetonitrile in the 1000–ml separatory funnel. The mixture was shaken 200 times and the phases were allowed to separate. The upper hexane was retained and washed twice with water to remove traces of acetonitrile. The hexane layer was drained into a 125–ml Erlen-

meyer flask containing a small quantity (2–3 g) of Na_2SO_4 which effectively removed the water. After standing for about an hour the hexane was transferred to a 250–ml Erlenmeyer flask with several rinses of hexane. A few crystals of Na_2SO_4 were added as boiling chips, a Snyder column was attached, and the solvent was removed by distillation on the steam bath.

FLORISIL COLUMN CHROMATOGRAPHY

Thirty g florisil containing 3 percent water was prepared for chromatographic separation by pre-elution with 50 ml acetone, 50 ml anhydrous diethyl ether, and finally with 100 ml dry nanograde hexane.

The pesticide extract in 2 ml hexane was carefully transferred to the florisil column with 3 to 4 1–ml portions of hexane; 250 ml 10 percent ether in hexane elutes aldrin, dieldrin, heptachlor, and heptachlor epoxide from the column. Eluates were collected in 500–ml Erlenmeyer flasks, Snyder columns were attached, and volume was reduced on a steam bath to approximately 5 ml. This was transferred to a 10–ml volumetric flask for gas chromatographic (GC) determination.

A model 204 Varian Aerograph gas chromatograph equipped with an electron-capture detector was used for dieldrin quantitation. Operating conditions were:

Column:	high resolution, 3.3 mm by 3 m, packed with 100/120-mesh Supelcoport containing 2.0 QF–1 and 1.25 percent OV–17
Detector:	electron-capture, ^{63}Ni
Temperatures:	injection port 225°C column 200°C detector 255°C
Carrier gas:	nitrogen, at a flow rate of 25 ml/min
Sensitivity:	10 picograms dieldrin caused a 10 percent reorder response with a noise level of approximately 1 percent

Results and Discussion

Residue levels of dieldrin in raw soybeans were higher in 1974 than in previous years (Table 5). This is in spite of the declining use of aldrin on Illinois cropland since 1967 (Table 1). Apparently the dieldrin level in Illinois is beginning a gradual decline and residues of dieldrin in raw soybeans probably will also decline; further monitoring of soybeans is needed to prove this theory. Soybeans from fields previously treated with aldrin had a higher residue of dieldrin than had soybeans from untreated fields. It is possible that dieldrin residues occur in raw soybeans from untreated fields due to the movement of dieldrin-contaminated soil particles by air and/or water from previously treated fields. In a study in Cincinnati, Ohio, Cohen and Pinkerton (*4*) reported the average monthly dust fall to be 15 tons/square mile. The dust, which originated in the southern high plains of Texas, contained 0.003 ppm

TABLE 5. *Dieldrin residues in soybeans by years in Illinois*

	All Fields		From Treated Fields [1]		From Untreated Fields [2]	
Year	No.	ppm	No.	ppm	No.	ppm
1965	88	0.0089	57	0.0101	31	0.0068
1966	95	0.0077	43	0.0119	52	0.0043
1967	52	0.0102	17	0.0187	35	0.0061
1971	106	0.0101	43	0.0165	63	0.0058
1974	72	0.0136	23	0.0190	49	0.0110

NOTE: Residues reported in average ppm.
[1] Soil treated with aldrin when field was in corn during previous years.
[2] Soil not treated with aldrin during previous years.

dieldrin. Authors further stated that soil particles are certain to move by air within a local area.

The percentage of fields with high dieldrin residues in soybeans has increased since the initial survey in 1965 (Table 6). In 1965 and 1966, no fields were found with residues above 0.03 ppm in soybeans. In 1967, 1.5 percent of the fields sampled had soybeans with a dieldrin residue above 0.03 ppm. In 1971, this increased to 7.1 percent of the fields and in 1974, to 8.4 percent. There is no official tolerance for dieldrin in soybeans so these residue levels are of concern.

Dieldrin residues in soybeans grown after corn varied greatly even where aldrin use was similar. This can be expected because of variation in soil, slope, and farmers' cultivation practices. Greatest variation occurred where aldrin had been applied as a row or furrow treatment. Authors assume that in some fields the soybean rows were spaced between the corn rows of the previous year, resulting in lower residues than would occur where the soybean rows coincided with the corn rows of the previous year. In one instance in 1971, soybeans from one field contained 0.07–0.08 ppm dieldrin, an extraordinarily high residue. The farmer had used only 1 pound of actual aldrin per acre for the 5 consecutive seasons from 1966 to 1970. The dieldrin residues in the soybeans were greater than expected. Detailed discussions revealed that the farmer had been meticulous in his application rates and had started planting each year precisely the same distance from the fence. A

TABLE 6. *Percent of fields with dieldrin residues in soybeans at various ranges in 1965, 1966, 1967, 1971, and 1974 in Illinois*

	Percent of Fields per Range						
Residue Range, ppm	1965	1966	1967	1971	1974	Avg.	No. Fields
0–0.01	67.8	72.5	58.5	58.8	52.8	64.0	264
0.01–0.02	31.1	20.6	29.2	24.7	26.3	26.3	109
0.02–0.03	1.1	6.9	10.8	9.4	12.5	7.3	30
0.03–0.04	0	0	1.5	5.9	4.2	1.2	5
0.04–0.05	0	0	0	0	4.2	0.9	4
0.05–0.06	0	0	0	0	0	0	0
0.06–0.07	0	0	0	0	0	0	0
0.07–0.08	0	0	0	1.2	0	0.3	1

narrow band of aldrin/dieldrin concentration was formed, resulting in an abnormally high pickup by the soybeans.

The area of Illinois in which the soybeans were grown showed no significant variations in the level of dieldrin residues present (Table 7).

Planting dates had no effect on the dieldrin residues in soybeans (Table 8).

Dieldrin residues in soybeans from fields with a history of aldrin application ranged from 0.0140 to 0.0201 ppm for different planting dates; no pattern was indicated.

Soybean varieties showed no apparent difference in dieldrin residue (Table 9).

Table 10 lists dieldrin levels in soybeans according to oil content. Data were insufficient to correlate dieldrin residues with oil content of the soybeans.

Conclusions

Residue levels of dieldrin in Illinois soybeans increased between 1965 and 1974.

The percent of fields which had soybeans with a dieldrin level above 0.03 ppm increased between 1965 and 1974.

Dieldrin residues in Illinois soybeans are expected to decline gradually now that use of aldrin has diminished and will soon cease.

No significant correlation was found between dieldrin levels in soybeans and the area of the State where they were grown, date of planting, or variety.

Acknowledgments

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TABLE 7. *Dieldrin residues in soybeans by districts in Illinois*

District	All Fields		From Treated Fields [1]		From Untreated Fields [2]	
	No.	ppm	No.	ppm	No.	ppm
Northwest	22	0.0093	12	0.0110	10	0.0072
Northeast	22	0.0123	15	0.0148	7	0.0069
West	36	0.0110	27	0.0122	9	0.0073
Central	36	0.0112	20	0.0137	16	0.0081
East	33	0.0101	17	0.0130	16	0.0069
West-southwest	33	0.0115	18	0.0148	15	0.0076
East-southeast	53	0.0095	23	0.0153	30	0.0050
Southwest	24	0.0054	7	0.0092	17	0.0038
Southeast	19	0.0080	10	0.0123	9	0.0032

NOTE: Residues reported in average ppm.
[1] Soil treated with aldrin when field was in corn during previous years.
[2] Soil not treated with aldrin during previous years.

TABLE 8. *Actual dieldrin residues in soybeans by planting date in Illinois*

Planting Date	All Fields		From Treated Fields [1]		From Untreated Fields [2]	
	No.	ppm	No.	ppm	No.	ppm
Up to May 10	30	0.0109	13	0.0165	17	0.0066
May 11–15	27	0.0114	10	0.0201	17	0.0063
May 16–20	23	0.0103	12	0.0146	11	0.0055
May 21–25	21	0.0098	8	0.0140	13	0.0073
May 26–30	21	0.0115	9	0.0194	12	0.0057
May 31–June 4	18	0.0094	5	0.0177	13	0.0062
June 5 and later	18	0.0062	3	0.0199	15	0.0035

NOTE: Residues reported in average ppm.
[1] Soil treated with aldrin when field was in corn during previous years.
[2] Soil not treated with aldrin during previous years.

TABLE 9. *Dieldrin residues in soybeans by variety in Illinois*

Variety	All Fields		From Treated Fields [1]		From Untreated Fields [2]	
	No.	ppm	No.	ppm	No.	ppm
Wayne	51	0.0124	21	0.0209	30	0.0065
Clark	24	0.0043	4	0.0087	20	0.0034
Mixtures	21	0.0080	7	0.0114	14	0.0063
Amsoy	13	0.0137	9	0.0171	4	0.0060
Harosoy	10	0.0127	4	0.0224	6	0.0061
Beeson	10	0.0101	6	0.0115	4	0.0080
Harosoy 63	6	0.0141	3	0.0204	3	0.0078
Clark 5	5	0.0045	0	–	5	0.0045
Shelby	5	0.0122	3	0.0128	2	0.0112
Hawkeye	3	0.0063	0	–	3	0.0063
0102	2	0.179	1	0.0197	1	0.0162
Adams	2	0.0127	1	0.0175	1	0.0078
Dare	2	0.0038	0	–	2	0.0038
0207	1	0.0066	0	–	1	0.0066
Hawkeye 63	1	0.0052	0	–	1	0.0052
Scott	1	0.0019	1	0.0019	0	–
Chippewa	1	0.0241	1	0.0241	0	–
Unknown	183	0.0083	100	0.0109	83	0.0052

NOTE: Residues reported in average ppm.
– = samples lost during shipment or analysis.
[1] Soil treated with aldrin when field was in corn during previous years.
[2] Soil not treated with aldrin during previous years.

TABLE 10. *Dieldrin residues and oil content of soybeans in Illinois*

Oil Content, %	Dieldrin Residues					
	All Fields		From Treated Fields [1]		From Untreated Fields [2]	
	No.	ppm	No.	ppm	No.	ppm
0.0–20.0 [3]	195	0.0083	102	0.0109	93	0.0053
20.0–21.0	23	0.0106	8	0.0194	15	0.0058
21.0–22.0	46	0.0121	19	0.0209	27	0.0059
22.0–23.0	41	0.0105	16	0.0167	25	0.0064
23.0–24.0	25	0.0091	12	0.0134	13	0.0051
24.0 and over	10	0.0052	3	0.0053	7	0.0052

[1] Soil treated with aldrin when field was in corn during previous years.
[2] Soil not treated with aldrin during previous years.
[3] Less than 20 percent or no data available. In most instances, oil content was unknown. This figure should be disregarded when comparing oil content and dieldrin residues.

recting the random selection of soybeans and for obtaining field histories of insecticide use and cropping practices.

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Preliminary Monitoring of Agricultural Pesticides in a Cooperative Tobacco Pest Management Project in North Carolina, 1971—First-Year Study [1]

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ABSTRACT

A preliminary tobacco pest management pesticide monitoring project was initiated in two counties of North Carolina in 1971 to develop an ecologically, economically, and socially acceptable system for protecting tobacco from damaging insect and disease pests. Results from the first year's study including sampling procedures, sample preparation, and analysis and residue data are presented in this paper.

Introduction

A cooperative tobacco pest management project was initiated in North Carolina in 1971 by the United States Department of Agriculture (USDA), North Carolina Department of Agriculture, and the North Carolina Agricultural Extension Service.

The pest management area was 145 square miles which contained 685 tobacco farms, 1369 tobacco fields, and 4,244 acres. Average tobacco acreage was 6.2 per farm and 3.1 per field.

This pilot study was an attempt to establish an ecologically, economically, and socially acceptable system for protecting tobacco crops from insect pests, as described by Ganyard et al. (2).

Another purpose of the preliminary study was to monitor pesticide residues in biotic and abiotic environmental components prior to application of the chemicals and following harvest of the crops, both within and outside program areas. Twenty sampling sites were selected in Wayne and Wilson Counties, ten of which were located within the pest management project area and ten in a geographically adjacent area. Each site consisted of a tobacco field with a farm pond located within 300 feet of the subject field and within the drainage area of the field. Ponds were reasonably uniform in size; duplicate samples were collected at each site according to standard sampling procedures.

Samples were collected in the spring before pesticide applications and again in the fall after pesticide treatments.

Biotic groups sampled in 1971 include bluegill (*Lepomis macrochirus*); four species of turtles, (a) snapping turtles (*Chelydra serpentina*), (b) musk turtles (*Sternotherus odoratus*), (c) painted turtles (*Chrysemys picta*), and (d) yellow-bellied turtles (*Chrysemys scripta*); large frogs (*Rana* sp); tiger beetles (*Megacephala carolina*); and cured tobacco leaves. Abiotic groups sampled were pond sediment, pond water, and tobacco field soil. Not all biotic groups were available from all sites, which accounts for occasional omissions of results from the tables.

SAMPLE COLLECTION

Turtles and fish were captured with a common fish trap constructed of chicken wire. Tiger beetles were captured in pitfall traps on the soil surface within each tobacco field. Traps were serviced daily and trappings for a given species were conducted simultaneously at all 20 sites. Bullfrogs were collected by gigging at night from a boat.

Water samples were collected from a boat by attaching a smallmouth, one-gallon bottle to a pole and slowly lowering it to the bottom of the pond. Ten samples were collected from dispersed positions within each pond, mixed thoroughly, and a one-gallon composite sample was taken for analysis.

Ten random sediment samples collected from each pond with an Eckman dredge were composited and a representative half-gallon sample was taken for analysis. A core sampling device was used to collect 50 soil cores, 2 in. by 5 in., from each tobacco field. Cores from each field were mixed thoroughly and a representative half-gallon sample was taken for analysis.

In a separate aspect of the study, cured tobacco leaves were randomly collected from field research plots. These samples were analyzed primarily for residue determinations following pesticide treatments with certain known chemicals.

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All biotic samples except tiger beetles were weighed in the field and placed in portable freezers containing dry ice. Tiger beetles were immobilized by cooling and returned to the laboratory where accurate weights were obtained on a Mettler balance. All samples except water were stored at 25°F until packed in styrofoam biomailers containing dry ice for shipment to the USDA Methods Development Environmental Quality Laboratory in Brownsville, Tex., for residue analysis. To avoid spoilage, samples were shipped airmail special delivery and normally arrived in Brownsville, still frozen, within 24 hours. Water samples were stored in one-gallon glass bottles and shipped to Brownsville approximately four days after collection. All samples except water were frozen until analysis. Water was stored in a walk-in cooler at approximately 40°F until analyzed. All samples were analyzed as soon as possible after receipt.

ANALYTICAL METHODS

Organochlorine and organophosphate pesticides were extracted from soil essentially as described by Stevens et al. (7); a brief description is given below.

Representative 300–g subsamples of wet soil were transferred to half-gallon Mason jars with 600 ml 3:1 mixture of hexane-isopropyl alcohol solvent and 80 ml distilled water, then rotated 4 hours on a concentric rotator. After the soil had settled, extracts were filtered through glass wool into 1-liter separatory funnels, the isopropanol and water were removed by washing three times with equal volumes of distilled water, and the hexane layer was filtered through a layer of glass wool and anhydrous sodium sulfate into 500–ml graduated cylinders, collecting 300–ml aliquots. The hexane extracts were then stored in capped, amber sample bottles at approximately 40°F pending gas chromatographic (GC) analysis.

Sediment was extracted in an identical manner with the following exceptions: excess water was drained from samples before subsamples were weighed and 250 g anhydrous sodium sulfate was added to the jars containing the solvent and samples.

After mixing water samples thoroughly, 500–g subsamples were extracted three times with 100-ml portions of nanograde dichloromethane in 1–liter separatory funnels. Extracts were drained through a glass wool/sodium sulfate filter into 500–ml Erlenmeyer flasks with glass heads and 1 ml 0.01 percent Nujol in hexane holding solution. Solvents were evaporated to approximately 5 ml through Snyder columns on a 60°C water bath, then 100 ml hexane was decanted through the Snyder columns slowly and the evaporation step was repeated. The concentrated extracts were transferred to 15–ml centrifuge tubes with nanograde hexane; solvent volumes were adjusted to 12.5 ml and stored in a 40°F refrigerator pending GC analysis.

Tobacco leaves were ground in a Hobart food chopper and 100–g subsamples were extracted in a half-gallon Mason jar with 800 ml acetonitrile by rotating concentrically at 30 rpm for 4 hours. Three-hundred-ml aliquots were collected through glass wool filters and transferred to 500–ml Erlenmeyer flasks. Acetonitrile was evaporated on hotplates at 95°F to approximately 10 ml and hexane was added and evaporated three times as described in the water extraction procedure. Finally, 100 ml hexane was added to the flask and transferred to 500–ml separatory funnels with 10 ml nanograde isopropyl alcohol and 10 ml hexane. The hexane layer was freed from water and alcohol by shaking with 200 ml distilled water and dried by filtering through anhydrous sodium sulfate into 250–ml Erlenmeyer flasks. Hexane was evaporated on a hot water bath; the final volume was adjusted to 100 ml and the extracts were stored in capped amber bottles pending cleanup.

Fish and frogs were processed whole; turtle shells were removed; composite samples were ground in a Hobart food chopper and 25–g subsamples were blended in a 1–liter blendor jar with 150 ml of a 3:1 mixture of nanograde hexane-isopropanol for 2 minutes. Blended samples were transferred to half-gallon Mason jars with 250 ml additional solvent and extracted as described previously. Extracts were filtered through glass wool into separatory funnels and washed three times with distilled water to remove alcohol. The hexane layer was filtered through sodium sulfate; aliquots were volumetrically measured and stored in amber bottles and refrigerated pending cleanup.

Tiger beetles (0.14–6.58 g) were macerated in 100–ml blendor jars with isopropanol for 2 minutes. Sample transfer to half-gallon jars was aided by two 75–ml rinsings of hexane. The extraction, filtering, drying, and storage procedures were identical to the previously described biological extracts.

Methomyl (Lannate) was extracted from soil, sediment, water and tobacco as described in a previous paper by Reeves and Woodham (6). Dichloromethane was used to extract the methomyl from soil, water, and sediment samples; a mixture of 97.5 percent dichloromethane and 2.5 percent benzene was employed for extracting the insecticide from tobacco samples. Extracts were extracted and stored as described previously for biological samples.

Carbaryl (Sevin) and carbofuran (Furadan) were extracted from soil, sediment, water, and tobacco as described by Reeves and Woodham (USDA Environmental Quality Laboratory, Brownsville, Tex., 1972: unpublished data). Briefly, the insecticides were extracted from sediment and tobacco with dichloromethane in half-gallon Mason jars on a concentric rotator for 4 hours, and water samples were extracted in separatory funnels with dichloromethane. Sample extracts were dried; the solvent was evaporated to 50 ml and refrigerated pending cleanup, hydrolysis, and derivation.

SAMPLE CLEANUP

Extracts from biological samples for organochlorine and organophosphate residue analyses were purified by partitioning 5–g aliquots between two immiscible solvents, hexane and acetonitrile, as described by Wiersma et al. (*8*). After concentrating the samples into hexane, a modification of the florisil column cleanup procedures of Mills et al. (*5*), Johnson (*4*), and Wiersma et al. (*9*) was used to further purfiy the extracts. The following changes were made: a mixture of 15 g florisil and 2 g anhydrous sodium sulfate was used in the columns; a third elution with dichloromethane removed more polar pesticides. Glass beads and 1 ml of a 0.01 percent Nujol in hexane solution were added and each fraction was concentrated through Snyder columns to approximately 5 ml on a hot water bath. The concentrated extracts were quantitatively transferred to stoppered 15–ml centrifuge tubes with hexane and the final solvent volume was adjusted to 12.5 ml. Care was taken to ensure that all diethyl ether or dichloromethane had been removed; then samples were refrigerated pending GC analysis.

Fraction 1 from the florisil columns contained pesticides of low polarity, such as aldrin, heptachlor, DDT, DDE, *o,p'*– and *p,p'*–TDE, toxaphene, strobane, chlordane, mirex, γ–BHC, and PCB's. Fraction 2 contained moderately polar pesticides such as dieldrin, endrin, heptachlor epoxide, methyl and ethyl parathion, endosulfan, and ethion. Fraction 3 contained the highly polar pesticides: malathion, methyl trithion, and endosulfan isomers.

Soil, sediment, water, and tobacco extracts were purified for methomyl analysis as described by Reeves and Woodham (*6*). Briefly, the method involved concentrating solvents to 10 ml, transferring the extracts into florisil chromatographic columns, and eluting from the columns with 90 percent dichloromethane : 10 percent acetone (v/v) for soil, sediment, and water. Water-saturated chloroform eluted methomyl from the florisil columns for the tobacco extracts. Following column cleanup, the eluates were evaporated and transferred to 15–ml centrifuge tubes for refrigeration pending GC analysis.

Soil, sediment, water, and tobacco extracts for carbaryl and carbofuran analysis were purified using a florisil chromatographic column cleanup procedure described by Reeves and Woodham, Brownsville, Tex., as cited earlier. The insecticides were eluted from columns with dichloromethane for soil, water, and sediment; for tobacco, 20 percent diethyl ether in dichloromethane was used. Eluates were evaporated, transferred to centrifuge tubes, and refrigerated pending hydrolysis, derivation, and coagulation.

The purified extracts were hydrolyzed with aqueous sodium hydroxide, then chloracetylated as described by Argauer et al. (*1*) to form derivatives detectable by electron-capture GC. The derivatized soil, sediment, and water samples were stored in benzene in centrifuge tubes with anhydrous sodium sulfate and analyzed by GC as soon as possible. The derivatized tobacco extracts were further purified by coagulation as described by Johnson (*3*). The derivatives were extracted from the aqueous phase with dichloromethane; the solvent was filtered through anhydrous sodium sulfate and evaporated to approximately 5 ml on a hot water bath. Extracts were transferred to centrifuge tubes with benzene and refrigerated pending GC analysis.

GAS CHROMATOGRAPHIC ANALYSIS

Organochlorine pesticides were analyzed by GC using a Tracor MT–200 gas chromatograph with the following instrument parameters and operating conditions:

Detectors:	dual Ni–63 electron-capture
Columns:	6–ft glass, packed with
(1)	3 percent DC–200 mesh Gas-Chrom Q
(2)	3 percent OV–1 on 80–100 mesh Chromosorb W
(3)	5 percent QF–1 on 100–120 mesh Gas-Chrom Q
(4)	mixture of 1.95 percent QF 1:1 and 5 percent OV–17 on 80–100 mesh Gas-Chrom Q
Temperatures:	column 200°C
	injector 225°C
	detector 300°C
Carrier gas:	nitrogen flowing at 80 ml/min; for mixed column, 120 ml/min

Recorder (1 mv) chart speed: 30 in./hr

Sensitivity was adjusted to produce approximately half-scale deflection of the recorder pen with 0.50–ng injection of aldrin. Calculations as ppm were based on heights of the pesticide peaks compared with a pesticide calibration standard of known concentration and purity.

Organophosphate pesticides were also analyzed with a Tracor MT–200 gas chromatograph equipped with a dual flame photometric detector (FPD) with the phosphorus (526 nm) and sulfur (394 nm) interference filters installed for simultaneous recordings of organophosphate and sulfur peaks from thiophosphate pesticides. Instrument parameters and operating conditions follow.

Column:	6-ft-by-1/4-in.-glass, packed with 3 percent DC–200 on 100–120 mesh Gas-Chrom Q
Temperatures:	column 200°C
	injector 225°C
	detector 200°C
Carrier gases:	nitrogen flowing at 120 ml/min
	air flowing at 80 ml/min
	hydrogen flowing at 200 ml/min
	oxygen flowing at 20 ml/min
Recorder chart speed:	30 in./hr

Sensitivity was adjusted to obtain approximately half-scale deflection with an injection of 1.5–ng ethyl parathion. Calculations were based on peak height as described for organochlorine pesticides.

Methomyl was analyzed as described in the organophosphate analysis section, except the column temperature was 140°C and a 10 percent DC–200 on 100–120 mesh Gas-Chrom Q column was used. Sensitivity of the detector was adjusted to obtain half-scale deflection of the recorder pen with a 20–ng injection of methomyl.

Carbaryl and carbofuran were analyzed by GC as described for the chlorinated hydrocarbons, except that the column

was 6-ft-by-1/4-in.-glass packed with 5 percent OV–210 on 100–120 mesh Chromosorb-W. Sensitivity was adjusted to obtain approximately half-scale deflection with a 4–ng injection of the carbofuran or carbaryl derivatives.

A series of controls consisting of a solvent check, nonfortified sample, and a sample fortified with known concentrations of pesticides were included with each group of samples. The controls were extracted, stored, and analyzed simultaneously with samples containing unknown concentrations of pesticides in order to monitor possible contamination and analytical and extraction efficiency. The following average range of recoveries was obtained from the various sample types: soil, 58.1 percent for aldrin to 117.0 percent for *p,p'*–DDT; sediment, 71.2 percent for dieldrin to 103.6 percent for *p,p'*–DDE; water, 58.9 percent for carbaryl to 98.4 percent for *p,p'*–DDE; tobacco, 47.6 percent for methyl trithion to 104.9 percent for ethion; frogs, 51.1 percent for malathion to 100.8 percent for *o,p'*–DDT; turtles, 55.8 percent for lindane to 89.7 percent for ethion. All residues were corrected for appropriate recovery values.

Moisture content of all soil and sediment samples was determined by drying a weighed 100–g sample in a 120°C oven for 24 hours, then reweighing the sample to determine moisture loss. Residues were corrected and reported on a dry-weight basis.

All pesticide residues were confirmed by partitioning coefficients (p–values), thin-layer chromatography, chemical derivatization, and multiple-column GC.

Results and Discussion

Table 1 presents pesticide treatment histories for the sampling sites in this project. Pesticides applied were either organophosphates or carbamates except the endosulfan treatments at sites F–3137 and G–1135.

Table 2 presents residue data for soil and sediment in areas inside and outside the program area. Residues ranging from 0.01 to 0.35 ppm *o,p'*–TDE and from 0.02 to 1.15 ppm *p,p'*–TDE were detected in sediment inside the program area. In sediment from ponds outside these areas, residues ranged from 0.01 to 0.30 ppm *o,p'*–TDE and from 0.02 to 0.87 ppm *p,p'*–TDE. No residues of organophosphates, carbaryl, methomyl (Lannate), or carbofuran (Furadan) exceeding the lower limits of sensitivity of 0.01 ppm or 0.05 ppm were detected in any sediment samples.

Generally, soil samples collected within the program area had slightly higher residues than had those outside the program area. Residues ranged from not detectable to 2.00 ppm carbofuran (Furadan). Soil from outside the program area revealed carbofuran residues up to 0.14 ppm, apparently from pesticide treatments in previous years.

TABLE 1. *Record of foliar insecticide treatments of tobacco in sampling sites in pest management areas of North Carolina, 1971* [1]

Sampling Site	Insecticides Applied
A–164	Guthion
A–167	Methomyl (Lannate)
C–541	Carbaryl (Sevin), endosulfan, malathion
C–559	No treatment
C–622	Carbaryl (Sevin)
C–1513	Guthion
D–822	Diazinon
E–3229	Carbaryl (Sevin)
E–3231	Guthion
F–1237	–
F–1281	No treatment
F–3137	Endosulfan, malathion
G–1135	Endosulfan, malathion
G–1348	Azodrin
G–1425	Methomyl (Lannate)
G–1508	Azodrin
G–1509 (site 1)	Carbaryl (Sevin)
G–1509 (site 2)	Azodrin
H–238	Parathion, carbaryl (Sevin)
H–256	Methomyl (Lannate)

[1] Ganyard et al., 1972 (2).

Of the biological samples, tiger beetles showed perhaps the greatest variety of pesticides. Inside and outside the program area, residues of dieldrin, endrin, and all six DDT isomers were detected. No significant differences occurred between samples from inside and those from outside the program area.

Most residues in biological samples from program areas were equal to or less than residues in samples collected outside these areas. Naturally there were some exceptions, such as *p,p'*–DDT in frogs (0.01 ppm outside and 0.02 ppm inside) and *p,p'*–DDE in tiger beetles (4.44 ppm inside and 3.07 ppm outside).

Table 3 shows residue patterns in tobacco samples from this pest management project area. Residues up to 0.31 ppm *o,p'*–TDE, 0.98 ppm *p,p'*–TDE, 0.28 ppm *o,p'*–DDT, and 1.91 ppm *p,p'*–DDT were detected. Carbofuran residues were found in samples from the following areas: Aycock No. 4, 0.96 ppm; Saul's No. 4, 0.47 ppm; R. Marsh No. 4, 0.08 ppm; and W. Harbor No. 4, 0.48 ppm. Carbofuran residues are not shown in the tables.

Residue data for turtles, frogs, fish, and insects within and outside the program area are presented in Table 4. Although measurable amounts of the chlorinated hydrocarbon pesticides were found, the quantities were not unusually high. The highest concentration was 3.81 ppm *p,p*–DDT in one turtle sample. No organophosphate residues exceeding the lower limits of 0.01 ppm were detected in any biological samples.

TABLE 2. *Organochlorine pesticide residues in soil and sediment from pest management areas in North Carolina, 1971*

Sampling Site	Sampling Series	Residue, ppm dry weight[1,2]					
		o,p'-DDE	*p,p'*-DDE	*o,p'*-TDE	*p,p'*-TDE	*o,p'*-DDT	*p,p'*-DDT
Inside Program Area							
SEDIMENT							
A-164	1	<0.01	<0.01	0.02	0.07	<0.01	<0.01
	2	<0.01	<0.01	0.03	0.16	<0.01	<0.01
A-167	1	<0.01	<0.01	0.35	1.15	<0.01	<0.01
	2	<0.01	<0.01	0.31	1.03	<0.01	<0.01
C-1135	1	<0.01	<0.01	0.05	0.09	<0.01	<0.01
	2	<0.01	<0.01	0.05	0.10	<0.01	<0.01
G-1348	1	<0.01	<0.01	0.05	0.20	<0.01	<0.01
	2	<0.01	<0.01	0.03	0.07	<0.01	<0.01
G-1425	1	<0.01	<0.01	0.08	0.15	<0.01	<0.01
	2	<0.01	<0.01	0.01	0.02	<0.01	<0.01
G-1508	1	<0.01	<0.01	0.01	0.04	<0.01	<0.01
	2	<0.01	<0.01	0.01	0.02	<0.01	<0.01
G-1509 (site 1)	1	<0.01	<0.01	0.10	0.21	<0.01	<0.01
	2	<0.01	<0.01	0.09	0.16	<0.01	<0.01
G-1509 (site 2)	1	<0.01	<0.01	0.08	0.24	<0.01	<0.01
	2	<0.01	<0.01	0.15	0.52	<0.01	<0.01
H-238	1	<0.01	<0.01	0.02	0.07	<0.01	<0.01
	2	<0.01	<0.01	0.04	0.07	<0.01	<0.01
H-256	1	<0.01	<0.01	0.04	0.15	<0.01	<0.01
	2	<0.01	<0.01	0.04	0.12	<0.01	<0.01
Outside Program Area							
C-541	1	<0.01	<0.01	0.05	0.15	<0.01	<0.01
	2	<0.01	<0.01	0.04	0.11	<0.01	<0.01
C-559	1	<0.01	<0.01	0.02	0.05	<0.01	<0.01
	2	<0.01	<0.01	0.03	0.08	<0.01	<0.01
C-622	1	<0.01	<0.01	0.02	0.05	<0.01	<0.01
	2	<0.01	<0.01	0.02	0.07	<0.01	<0.01
C-1513	1	<0.01	<0.01	0.02	0.07	<0.01	<0.01
	2	<0.01	<0.01	0.01	0.04	<0.01	<0.01
D-822	1	<0.01	<0.01	0.02	0.02	<0.01	<0.01
	2	<0.01	<0.01	0.01	0.05	<0.01	<0.01
E-3229	1	<0.01	<0.01	0.03	0.08	<0.01	<0.01
	2	<0.01	<0.01	0.03	0.07	<0.01	<0.01
E-3231	1	<0.01	<0.01	0.01	0.03	<0.01	<0.01
	2	<0.01	<0.01	0.03	0.07	<0.01	<0.01
F-1237	1	<0.01	<0.01	0.30	0.87	<0.01	<0.01
	2	<0.01	<0.01	0.19	0.48	<0.01	<0.01
F-1281	1	<0.01	<0.01	0.09	0.16	<0.01	<0.01
	2	<0.01	<0.01	0.05	0.10	<0.01	<0.01
F-3137	1	<0.01	<0.01	0.06	0.13	<0.01	<0.01
	2	<0.01	<0.01	0.05	0.14	<0.01	<0.01
Inside Program Area							
SOIL							
A-164	1	<0.01	<0.01	0.03	0.04	0.04	0.07
A-167	1	<0.01	<0.01	0.05	0.19	0.17	0.50
G-1135	1	<0.01	<0.01	0.03	0.11	0.04	0.12
G-1348	1	<0.01	<0.01	0.05	0.14	0.06	0.54
G-1425	1	<0.01	<0.01	0.04	0.08	0.03	0.07
G-1508	1	<0.01	<0.01	0.01	0.04	0.02	0.06
G-1509 (site 1)	1	<0.01	<0.01	0.02	0.06	0.03	0.09
G-1509 (site 2)	1	<0.01	<0.01	0.03	0.09	0.05	0.14
H-238	1	<0.01	<0.01	0.03	0.08	0.07	0.17
H-256	1	<0.01	<0.01	0.02	0.04	0.03	0.08
Outside Program Area							
C-5541	1	<0.01	<0.01	0.03	0.08	0.02	0.04
C-559	1	<0.01	<0.01	0.04	0.06	0.04	0.09
C-622	1	<0.01	<0.01	0.04	0.06	0.02	0.05
C-1513	1	<0.01	<0.01	0.07	0.14	0.04	0.10
D-822	1	<0.01	<0.01	0.02	0.04	0.03	0.05
E-3229	1	<0.01	<0.01	0.04	0.07	0.01	0.04
F-1237	1	<0.01	<0.01	0.06	0.13	0.02	0.04
F-1281	1	<0.01	<0.01	0.02	0.07	0.03	0.06
F-3137	1	<0.01	<0.01	0.02	0.04	0.10	0.14

[1] Corrected for pesticide recovery from fortified samples.
[2] Lower limits of sensitivity = 0.01 ppm.

TABLE 3. *Organochlorine pesticide residues in tobacco from pest management areas in North Carolina, 1971* [1]

Sampling Site	Residue, ppm dry weight [2,3]					
	o,p'-DDE	*p,p'*-DDE	*o,p'*-TDE	*p,p'*-TDE	*o,p'*-DDT	*p,p'*-DDT
Saul, No. 1	<0.01	<0.01	0.09	0.04	0.03	0.22
Saul, No. 2	<0.01	<0.01	0.03	0.06	0.04	0.39
Saul, No. 3	<0.01	<0.01	0.02	0.05	0.04	0.27
Ward, No. 1	<0.01	<0.01	0.31	0.69	0.28	1.91
Ward, No. 2	<0.01	<0.01	0.23	0.58	0.24	1.90
Ward, No. 3	<0.01	<0.01	0.15	0.54	0.17	1.83
Wilson, No. 1	<0.01	<0.01	0.02	0.05	0.04	0.20
Wilson, No. 2	<0.01	<0.01	0.02	0.04	0.04	0.19
Wilson, No. 3	<0.01	<0.01	0.03	0.05	0.05	0.23
Aycock, No. 1	<0.01	<0.01	0.04	0.12	0.04	0.25
Aycock, No. 2	<0.01	<0.01	0.02	0.05	0.03	0.15
Aycock, No. 3	<0.01	<0.01	0.03	0.98	0.03	0.21
Aycock, No. 4	<0.01	<0.01	0.02	0.06	0.04	0.18
W. Saul's	<0.01	<0.01	<0.01	<0.01	<0.01	0.22
W. Harbor, No. 1	<0.01	<0.01	<0.01	<0.01	<0.01	0.20
W. Harbor, No. 3	<0.01	<0.01	<0.01	<0.01	<0.01	0.23
W. Harbor, No. 4	<0.01	<0.01	<0.01	<0.01	<0.01	0.20
R. Marsh, No. 1	<0.01	<0.01	<0.01	<0.01	<0.01	0.07
R. Marsh, No. 2	<0.01	<0.01	<0.01	<0.01	<0.01	0.09
R. Marsh, No. 3	<0.01	<0.01	<0.01	<0.01	<0.01	0.09
R. Marsh, No. 4	<0.01	<0.01	<0.01	<0.01	<0.01	0.09

[1] Samples collected at harvest in autumn 1971.
[2] Corrected for pesticide recovery from fortified samples.
[3] Lower limits of sensitivity = 0.01 ppm.

TABLE 4. *Organochlorine pesticide residues in biological samples from pest management areas in North Carolina, 1971* [1]

Sampling Site	Species	Residue, ppm [2,3]							
		Dieldrin	Endrin	*o,p'*-DDE	*p,p'*-DDE	*o,p'*-TDE	*p,p'*-TDE	*o,p'*-DDT	*p,p'*-DDT
		Inside Program Area							
TURTLES									
A–164	Snapping	<0.01	<0.01	<0.01	0.29	<0.01	<0.01	0.01	<0.01
A–167	Yellow-Bellied	<0.01	<0.01	<0.01	L[4]	<0.01	0.02	0.01	0.04
G–1135	Mush	<0.01	<0.01	<0.01	0.11	<0.01	<0.01	0.01	<0.01
G–1135	Yellow-Bellied	<0.01	<0.01	<0.01	0.04	<0.01	<0.01	0.01	<0.01
G–1348	Yellow-Bellied	<0.01	<0.01	<0.01	0.14	<0.01	0.01	0.01	0.02
G–1425	Painted	<0.01	<0.01	<0.01	0.05	<0.01	<0.01	0.01	<0.01
G–1425	Yellow-Bellied	<0.01	<0.01	<0.01	0.03	<0.01	<0.01	0.01	<0.01
G–1425	Snapping	<0.01	<0.01	<0.01	0.08	<0.01	<0.01	0.01	<0.01
G–1425	Musk	<0.01	<0.01	<0.01	0.33	<0.01	<0.01	0.01	<0.01
G–1508	Yellow-Bellied	<0.01	<0.01	<0.01	0.08	<0.01	<0.01	0.01	0.02
G–1509	Musk	<0.01	<0.01	<0.01	0.21	<0.01	<0.01	0.01	<0.01
G–1509	Yellow-Bellied	<0.01	<0.01	<0.01	0.12	<0.01	<0.01	0.01	<0.01
H–238	Painted	<0.01	<0.01	<0.01	0.09	<0.01	<0.01	0.01	<0.01
H–238	Yellow-Bellied	<0.01	<0.01	<0.01	0.24	<0.01	0.01	0.01	0.04
H–256	Musk	<0.01	<0.01	<0.01	0.78	<0.01	<0.01	0.01	<0.01
H–256	Painted	<0.01	<0.01	<0.01	1.14	<0.01	0.02	0.01	0.04
H–256	Yellow-Bellie	<0.01	<0.01	<0.01	0.20	<0.01	0.02	0.01	0.03
		Outside Program Area							
C–541	Musk	<0.01	<0.01	<0.01	0.40	<0.01	<0.01	<0.01	<0.01
C–541	Painted	0.01	0.01	<0.01	0.34	<0.01	0.01	<0.01	0.02
C–559	Snapping	<0.01	<0.01	<0.01	0.27	<0.01	<0.01	<0.01	<0.01
C–622	Snapping	<0.01	<0.01	<0.01	0.11	<0.01	<0.01	<0.01	<0.01
C–1513	Musk	<0.01	<0.01	<0.01	0.33	<0.01	<0.01	<0.01	<0.01
C–1513	Painted	<0.01	<0.01	<0.01	0.40	<0.01	0.01	<0.01	0.01
D–822	Snapping	<0.01	<0.01	<0.01	0.18	<0.01	<0.01	<0.01	<0.01
D–822	Musk	<0.01	<0.01	<0.01	0.26	<0.01	<0.01	<0.01	<0.01
D–822	Painted	<0.01	<0.01	<0.01	0.13	<0.01	0.01	<0.01	<0.01
D–822	Yellow-Bellied	<0.01	<0.01	<0.01	0.03	<0.01	<0.01	<0.01	<0.01
E–3229	Painted	<0.01	<0.01	<0.01	0.26	<0.01	0.01	<0.01	0.01
E–3229	Snapping	<0.01	<0.01	<0.01	0.04	<0.01	<0.01	<0.01	<0.01
E–3229	Yellow-Bellied	<0.01	<0.01	<0.01	0.11	<0.01	0.01	<0.01	0.01
E–3231	Musk	<0.01	<0.01	<0.01	0.35	<0.01	<0.01	<0.01	<0.01
E–3231	Painted	<0.01	<0.01	<0.01	0.75	<0.01	0.01	<0.01	0.06
E–3231	Snapping	<0.01	<0.01	<0.01	0.15	<0.01	<0.01	<0.01	<0.01
E–3231	Yellow-Bellied	<0.01	<0.01	<0.01	0.70	<0.01	0.01	<0.01	0.01
F–1237	Musk	<0.01	<0.01	<0.01	3.81	<0.01	<0.01	<0.01	<0.01
F–1237	Painted	<0.01	<0.01	<0.01	1.99	<0.01	0.08	<0.01	<0.01
F–1237	Yellow-Bellied	<0.01	<0.01	<0.01	1.99	<0.01	0.08	<0.01	0.07

(Continued next page)

TABLE 4 (cont'd.). *Organochlorine pesticide residues in biological samples from pest management areas in North Carolina, 1971*[1]

Sampling Site	Species	Residue, ppm[2,3]							
		Dieldrin	Endrin	*o,p'*-DDE	*p,p'*-DDE	*o,p'*-TDE	*p,p'*-TDE	*o,p'*-DDT	*p,p'*-DDT
Inside Program Area									
F-1281	Musk	<0.01	<0.01	<0.01	0.17	<0.01	<0.01	<0.01	<0.01
F-1281	Painted	<0.01	<0.01	<0.01	0.18	<0.01	<0.01	<0.01	<0.01
F-1281	Snapping	<0.01	<0.01	<0.01	0.20	<0.01	<0.01	<0.01	<0.01
F-3137	Painted	<0.01	<0.01	<0.01	1.08	<0.01	0.01	<0.01	0.05
Inside Program Area									
FROGS									
A-164		<0.01	<0.01	<0.01	0.04	<0.01	<0.01	<0.01	<0.01
A-167		<0.01	<0.01	<0.01	0.04	<0.01	<0.01	<0.01	<0.01
C-622		<0.01	<0.01	<0.01	0.03	<0.01	<0.01	<0.01	<0.01
G-1135		<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01
G-1508		<0.01	<0.01	<0.01	0.10	<0.01	<0.01	<0.01	<0.01
G-1509		<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01
H-256		0.05	<0.01	0.01	0.14	<0.01	0.13	<0.01	0.01
Outside Program Area									
C-1513		<0.01	<0.01	<0.01	0.02	<0.01	<0.01	<0.01	<0.01
D-822		<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
E-3229		0.01	<0.01	<0.01	0.06	<0.01	<0.01	<0.01	<0.01
E-3231		<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01
F-1237		0.11	<0.01	0.02	0.15	<0.01	0.33	<0.01	0.02
F-1281		<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01
Inside Program Area									
FISH									
A-164	Bluegills	0.02	<0.01	0.04	0.19	<0.01	0.15	<0.01	0.03
A-167	Bluegills	0.01	<0.01	0.15	0.87	0.02	0.62	<0.01	0.09
G-1135	Bluegills	0.02	<0.01	0.05	0.11	0.01	0.14	<0.01	0.02
G-1348	Bluegills	<0.01	<0.01	0.03	0.11	<0.01	0.06	<0.01	0.02
G-1425	Bluegills	0.08	<0.01	0.03	0.07	<0.01	0.06	<0.01	0.02
G-1508	Bluegills	0.02	<0.01	0.02	0.16	<0.01	0.05	<0.01	0.04
G-1509	Bluegills	0.01	<0.01	0.03	0.14	<0.01	0.09	<0.01	0.02
H-238	Bluegills	0.01	<0.01	0.03	0.16	<0.01	0.07	<0.01	0.04
H-256	Bluegills	0.16	<0.01	0.09	0.12	0.01	0.15	0.01	0.04
Outside Program Area									
C-541	Bluegills	<0.01	<0.01	0.03	0.10	<0.01	0.05	0.01	0.01
C-559	Bluegills	<0.01	<0.01	0.01	0.09	<0.01	0.02	<0.01	<0.01
C-622	Bluegills	<0.01	<0.01	0.01	0.06	<0.01	0.02	<0.01	0.01
G-1513	Bluegills	<0.01	<0.01	0.06	0.15	<0.01	0.06	0.09	0.03
D-822	Bluegills	<0.01	<0.01	0.02	0.09	<0.01	0.06	<0.01	0.02
E-3229	Bluegills	0.01	<0.01	0.03	0.15	<0.01	0.07	<0.01	0.01
E-3231	Bluegills	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01
F-1237	Bluegills	0.11	<0.01	0.02	0.15	<0.01	0.33	<0.01	0.02
F-1281	Bluegills	0.06	<0.01	0.12	0.80	<0.01	0.32	0.02	0.07
F-3137	Bluegills	0.16	<0.01	0.05	0.12	0.01	0.19	0.01	0.04
G-1435	Bluegills	0.05	<0.01	0.04	0.08	<0.01	0.06	<0.01	0.01
Inside Program Area									
TIGER BEETLES									
A-164		<0.01	0.01	0.11	1.80	<0.01	0.01	0.04	<0.01
A-167		0.01	0.01	0.04	1.02	<0.01	0.02	0.05	0.02
F-1281		0.05	0.05	0.04	1.26	<0.01	0.01	0.06	0.01
F-3137		<0.01	0.02	0.06	2.02	0.01	0.02	0.06	0.03
F-3231		<0.01	<0.01	<0.14	2.44	<0.01	<0.01	0.12	0.01
G-1135		<0.01	<0.01	<0.01	1.58	<0.01	<0.01	0.01	<0.01
G-1348		<0.01	<0.01	<0.01	4.44	<0.01	<0.01	0.01	<0.01
G-1425		<0.01	<0.01	<0.01	0.91	<0.01	<0.01	0.01	<0.01
G-1508		<0.01	<0.01	<0.01	1.38	<0.01	<0.01	0.01	<0.01
G-1509	(site 1)	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	0.01	<0.01
G-1509	(site 2)	<0.01	<0.01	<0.01	2.62	<0.01	<0.01	0.01	<0.01
H-238		<0.01	<0.01	<0.01	2.10	<0.01	0.01	0.01	0.03
H-256		0.04	<0.01	0.01	0.06	0.01	0.01	0.01	0.03

(Continued next page)

TABLE 4 (cont'd.). *Organochlorine pesticide residues in biological samples from pest management areas in North Carolina, 1971* [1]

Sampling Site	Species	Residue, ppm [2,3]							
		Dieldrin	Endrin	*o,p'*-DDE	*p,p'*-DDE	*o,p'*-TDE	*p,p'*-TDE	*o,p'*-DDT	*p,p'*-DDT
Inside Program Area									
Outside Program Area									
C–541		<0.01	<0.01	<0.01	0.46	<0.01	<0.01	<0.01	<0.01
C–559		<0.01	<0.01	<0.01	3.07	<0.01	<0.01	<0.01	<0.01
C–622		<0.01	<0.01	<0.01	1.88	<0.01	<0.01	<0.01	<0.01
G–1513		<0.01	<0.01	<0.01	1.86	<0.01	<0.01	<0.01	<0.01
E–3229		<0.01	<0.01	<0.01	1.03	<0.01	<0.01	<0.01	<0.01
E–3231		<0.01	<0.01	0.14	2.44	<0.01	<0.01	0.12	0.01
E–3231		<0.01	<0.01	<0.01	1.37	<0.01	<0.01	<0.01	<0.01
F–1237		<0.01	<0.01	<0.01	2.39	<0.01	<0.01	<0.01	<0.01
F–1281		0.05	0.05	0.04	1.26	<0.01	0.01	0.06	0.01
F–3137		0.01	0.02	0.06	2.02	<0.01	0.02	0.06	0.03

[1] Samples collected at beginning of crop season.
[2] Corrected for pesticide recovery from fortified samples.
[3] Lower limits of sensitivity = 0.01 ppm.
[4] L. Lost samples.

Conclusions

This one-year study did not produce evidence that pesticide use in Wayne and Wilson Counties is causing significant residue accumulation in the environment. Chemicals used for tobacco pest control are very similar throughout North Carolina, varying only in frequency of use. To project statewide trends and draw conclusions concerning pest management in tobacco and other crops, additional residue information must be accumulated. Plans are in progress to continue these studies for several years, establishing a more reliable baseline for projecting trends.

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APPENDIX

Chemical Names of Compounds Discussed in This Issue

ALDRIN	Not less than 95% of 1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4: 5,8-dimethanonaphthalene
AROCLOR	A mixture of chlorinated terphenyls
AZINPHOSMETHYL	*0,0*-dimethyl *S*[4-oxo-1,2,3,-benzotriazin-3(4H)ylmethyl] phosphorodithioate
AZODRIN	3-hydroxy-*n*-methyl-*cis*-crotonamide, dimethyl phosphate
BHC(Benzene Hexachloride)	1,2,3,4,5,6-hexachlorocyclohexane (mixture of isomers). Commercial product contains several isomers of which *gamma* is most active as an insecticide.
CARBARYL	1-naphthalenyl methylcarbamate
CARBOFURAN	2,3-dihydro-2,2-dimethyl-7-benzofuranyl methylcarbamate
CHLORDANE	1,2,3,5,6,7,8,8-octachloro-2,3,3a, 4,7,7a-hexahydro-4,7-methanoindene. The technical product is a mixture of several compounds, including heptachlor, chlordene, and two isomeric forms of chlordane.
DDE	Dichlorodiphenyl dichloro-ethylene (degradation product of DDT) *p,p'*–DDE: 1,1-Dichloro-2,2-bis(*p*-chlorophenyl) ethylene *o,p'*–DDE: 1,1-Dichloro-2-(*o*-chlorophenyl)-2-(*p*-chlorophenyl) ethylene
DDT	Main component (*p,p'*–DDT):α-Bis(*p*-chlorophenyl) β,β,β-trichloroethane. Other isomers are possible and some are present in the commercial product. *o,p'*–DDT: [1,1,1-Trichloro-2(*o*-chlorophenyl)-2(*p*-chlorophenyl) ethane]
DIELDRIN	Not less than 85% of 1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-*endo-exo*-5,8-dimethanonaphthalene
ENDOSULFAN	6,7,8,9,10,10-Hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3-benzodioxathiepin 3-oxide
ENDRIN	1,2,3,4,10,10-Hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-*endo-endo*-5,8-dimethanonaphthalene
ETHION	0,0,0',0'-Tetraethyl S,S'-methylene bisphosphorodithioate
ETHYL PARATHION	*0,0*-diethyl-*o-p*-nitrophenyl phosphorothioate
GUTHION	See azinphosmethyl.
HEPTACHLOR	1,4,5,6,7,8,8-heptachloro-3a,4,7,7a-tetrahydro-4,7-*endo*-methanoindene
HEPTACHLOR EPOXIDE	1,4,5,6,7,8,8-heptachloro 2,3-epoxy-3a,4,7,7a-tetrahydro-4,7 methanoindane
LINDANE	Gamma isomer of benzene hexachloride (1,2,3,4,5,6-hexachlorocyclohexane) of 99+ % purity
MALATHION	S-[1,2-Bis(ethoxycarbonyl) ethyl] 0,0-dimethyl phosphorodithioate
METHOMYL	methyl *N*-[[(methylamino) carbonyl] oxy] ethanimidothioate
METHYL PARATHION	O,O-Dimethyl O-*p*-nitrophenyl phosphorothioate
METHYL TRITHION	O,O-Dimethyl S-(*p*-chlorophenylthio) methyl phosphorodithioate
MIREX	1,1a,2,2,3,3a,4,5,5,5a,5b,6-dodecachlorooctahydro-1,3,4-metheno-1H-cyclobuta[cd]pentalene
PCB's (Polychlorinated Biphenyls)	Mixtures of chlorinated biphenyl compounds having various percentages of chlorine
STROBANE	Polychlorinates of camphene, pinene, and related terpenes
TDE	2,2-Bis(*p*-chlorophenyl)-1,1-dichloroethane
TOXAPHENE	Chlorinated camphene (67-69% chlorine). Product is a mixture of polychlor bicyclic terpenes with chlorinated camphenes predominating.

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